Microstructure and Mechanical Properties of Dual Phase Steels, with Different Martensite Morphology, Produced During TLP Bonding of a Low C-Mn Steel

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In this research, production of ferrite - martensite dual phase steels with different martensite morphology was considered during transient liquid phase bonding of a low carbon steel. The steel was bonded using an iron base interlayer with melting point of 1443 K and 40 μm thickness. Bonding process carried out at 1473 K, under pressure of 0.5 MPa, at different holding time of 10, 20, 30 and 40 minutes. Microstructural studies of joint region showed that isothermal solidification completed at the bonding time of 40 minutes. Microstructure of joints made at the bonding time of 10, 20, and 30 minutes consists of two distinct region, athermal and isothermal solidified zones. Microstructure of these zones was studied and chemical composition of these zones was determined by EDS. Joints made with bonding time of 40 minutes were homogenized at 1008 K and then cooled into cold water to produce dual phase ferrite and martensite microstructure with different martensite morphology. According to shear test results, it was found that the shear strength of ferrite - fibrous martensite microstructure is greater than those with ferrite - continuous martensite and ferrite - blocky martensite microstructure.

Keywords: transient liquid phase bonding, solidification, homogenization, mechanical properties, microstructure

1. INTRODUCTION

Transient liquid phase (TLP) bonding is a hybrid process of brazing and solid-state diffusion bonding which is extensively used to join superalloys, ceramics, and complex alloys [1,2]. In this technique an interlayer, which contains melting point depressant (MPD) elements (e.g. B, P, and Si), is inserted between two joining components surfaces and heated to above the interlayer melting point temperature. The holding time at the bonding temperature should be sufficient to complete isothermal solidification of the joint due to the compositional changes between the melted interlayer and parent alloy [3,4]. After completion of the isothermal solidification, the bonding process continues with a solid state homogenization at a suitable temperature [5].

Ferrite - martensite dual phase steels (DP) are subset of advanced high strength steels which widely used in transportation industries, oil and gas pipelines, pressurized tanks, high voltage power masts, and iron bridges because of their unique characteristics such as high deformability with high strength, high initial strain hardenability, low ratio of yield strength to ultimate tensile strength, resistance to high fatigue levels, good weldability, and impact resistance [6,7]. Mechanical properties of DP steels are mainly controlled by martensite volume fraction, carbon content of martensite, and martensite morphology [8,9].

Three different methods, inter-critical quenching (ICQ), inter-mediate quenching (IMQ), and step quenching (SQ), are used to produce DP steels with different martensite morphology [10].

In the ICQ method, homogenized steel with (α+pearlite) microstructure is heated to the inter-critical temperature at the (α+γ) region and held for adequate time. Thus austenite grains nucleate and grow at the grain boundaries of ferrite, and form an austenitic continuous network which eventually transforms to martensite with subsequent quenching from inter-critical temperature [7,10,11]. The obtained microstructure consists of coaxial ferrite grains surrounded by martensitic continuous network that known as ferrite - continuous martensite (FCM) DP steel.

In the IMQ method, austenitized steel quenched into water which results in fully martensitic microstructure. The Quenched steel is then heated to the inter-critical temperature and quenched in water again after sufficient holding time at this temperature [10,12]. The obtained microstructure is named ferrite - fibrous
martensite (FFM) DP steel.

In the SQ method, the steel is austenitized and cooled to inter-critical temperature from austenitization temperature and quenched then in to water after sufficient holding time at this temperature. During this heat treatment cycle, the ferrite grains nucleate at the austenite grain boundaries and grow and then retained austenite transforms to martensite during quenching [7,10]. The resultant microstructure consists of large blocky shaped martensite islands surrounded by coarse ferrite matrix which is known as ferrite - blocky martensite (FBM) DP steel.

With respect to DP steels applications, welding of these steel is also important. The most common methods used for DP steels welding include: resistance welding, arc welding, laser welding and solid state welding. The thermal process of this welding methods leads to inhomogeneous microstructure and mechanical properties of the weld zone in comparison to parent alloy [6, 13-15]. Recently, producing of ferrite – martensite DP steel during the TLP bonding process has been reported [16]. However the effect of martensite morphology on mechanical properties of DP steels produced during TLP bonding process has not been studied yet. The aim of the present research is producing DP steels with different martensite morphology during TLP bonding process. Also the effect of martensite morphologies on microstructure and mechanical properties of the joint region were also investigated.

2. EXPERIMENTAL PROCEDURES

2.1. Materials

In this study, the hot-rolled St52 steel plate with 12 mm thickness was used as the initial material. For bonding process, an iron base interlayer with melting point of about 1443 K and 40 μm thickness was used. Chemical composition of St52 and the interlayer is given in Table 1.

2.2. Experimental procedures

To make the bonding process and DP steel production heat treatment cycle simultaneously, bonding process was done simultaneously with austenitizing of steel and bond homogenization carried out at the inter-critical temperature to produce DP steels with different martensite morphology. For bonding process, both steel and interlayer samples were cut with dimensions of 12 × 12 × 6 mm³ and 12 × 12 × 3 mm³ respectively. Contact surfaces of steel were ground using 1000 grade SiC paper and ultrasonically cleaned and stored in methanol until being used for bonding. Bonding process carried out at 1473 K, above the melting point of interlayer, using induction furnace and applied pressure of 0.5 MPa at different holding times. The required bonding time for completion of isothermal solidification was chosen as the optimum time of bonding. As will discuss in the result section this time was 40 minutes.

The low and high critical temperatures of (α+γ) region of used steel were calculated according to the experimental equations (1) and (2) [7]. These temperatures are 997 K and 1115 K respectively. Therefore, the temperature of 1008 K was considered as the inter-critical temperature for DP steels production, or bond homogenization. After determination of optimum bonding time at 1473 K, various heat treatment cycles were tested to produce DP steels with different martensite morphology, but with the same volume fraction of martensite about 40%. Table 2 shows heat treatment cycles for bonding, at optimum bonding time, and dual phase steel producing processes or bond homogenization.

\[
A_{c1} (K) = 996 - 10.7Mn - 16.9Ni + 29.1Si + 16.9Cr + 290As + 6.38W (1)
\]

\[
A_{c3} (K) = 1183 - 203\sqrt{C} - 15.2Ni + 44.7Si + 104V + 31.5Mo + 13.1W (2)
\]

Joints microstructure was studied by optical and scanning electron microscopes (SEM) on prepared metallographic specimens etched with Nital 2%. Chemical compositions of joint region were determined by energy dispersive spectroscopy (EDS).

Micro-hardness measurements were taken across the joint region with a load of 50gr, according to ASTM E384 [17]. Shear tests were performed with a tensile machine at a cross-head speed of 1 mm/min, according to ASTM D1002 [18] on samples with dimensions of 10 × 10 × 10 mm³ using a fixture (Fig. 1). For bonded and homogenized samples, three samples

| Table 1. Chemical compositions of used steel and interlayer (wt%) |
|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Element        | C   | Si  | Mn  | P   | S   | Fe  | Ni  | Mo  | B   |
| Steel          | 0.19| 0.44| 1.3 | 0.018| 0.13| Base| -   | -   | -   |
| Interlayer     | -   | -   | -   | -   | -   | 44.3| 44.23| 7.61| 3.86|

| Table 2. Heat treatment cycles for TLP bonding process or DP steel producing with different martensite morphology |
|-----------------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Auestenitzing or bonding condition      | Cooling from bonding condition | Dual phase producing or bond homogenization condition | Cooling from homogenization condition | Microstructure  |
| 1473 K - 40 min                        | Cooling in air   | 1008 K - 60 min | Quenching into cold water | FCM            |
| 1473 K - 40 min                        | Quenching in cold water | 1008 K - 75 min | Quenching into cold water | FFM            |
| 1473 K - 40 min                        | Cooling to 1008K | 1008 K - 80 min | Quenching into cold water | FBM            |
were tested and the average was taken as the shear strength of bonded sample. Base metal shear tests specimens were heat treated at the same heat treatment cycle of bonded samples (Table 2) to produce the same microstructure, i.e. FCM, FFM, and FBM microstructure.

3. RESULTS AND DISCUSSION

3.1. Microstructure

Figure 2 shows microstructures of DP steels, produced according to Table 2, with different martensite morphology and martensite volume fraction of about 40%.

To get the required time of complete isothermal solidification during TLP bonding at 1473 K, bonding process carried out at different holding time and the bonded samples were then cooled in air. Sections of bonded samples were prepared to study the joint microstructure.

Figure 3 shows microstructure of joint made at 1473 K for different holding time of 10, 20, 30 and 40 minutes. As can be seen, the joint band (JB) of bonds made at the holding times of 10, 20 and 30 minutes consists of two distinct zones, athermally solidified zone (ASZ), and isothermally solidified zone (ISZ). But with increasing the bonding time, the ISZ increases and ASZ decreases and disappears at the holding time of 40 minutes. This means complete isothermal solidification happened at 40 minutes holding time and this time is the optimum time for bonding of used steel at 1473 K. In the TLP process, by heating the samples to bonding temperature the interlayer melts and holding the samples at this temperature causes interdiffusion of alloying elements between the base material and the liquid. Diffusion of boron from the liquid into the base...
metal increases the melting point of liquid and isothermal solidification begins at the liquid/solid interface. If the holding time is not sufficient for complete isothermal solidification, the remained liquid at the bonding temperature transforms and ASZ formed during cooling from the bonding temperature ($a$-$3c$).

For example, Fig. 4 shows the BS-SEM photograph of joint made at 1473 K for 30 min. In this figure the ISZ is shown as C and ASZ phases as A and B. EDS point analysis of phases A, B, and C is given in Fig. 5 and Table 3. Although the EDS analysis is not able to measure the boron and carbon contents accurately but it can shows the presence of these elements and their relative values (Fig. 5). Therefore, the boron and carbon contents are not reported in Table 3. Figure 5 shows that during the bonding process, alloying elements diffusion occurred between melted interlayer and base metal.

The isothermal section of Fe-Ni-Mo ternary diagram at 1473 K is shown in Fig. 6 [19]. According to this figure and Fe, Ni, and Mo concentration (Table 3), the ISZ (C phase) is a solid solution of Ni in $\gamma$-Fe at the bonding temperature. Based on Fe-Mo binary diagram phase, the limit solubility of Mo in $\gamma$-Fe is very low [19].

Figure 5 shows that the amount of Mo in ASZ (phases A and B) is considerably more than that in phase C. This means that with progressing of isothermal solidification at 1473 K, Mo rejected into the remained liquid. There is no liquid phase in isothermal section of Fe-Ni-Mo ternary diagram at 1473 K (Fig. 6), therefore the presence of liquid phase at 1473 K in joint bands of bonds made at the holding time of 30 minutes and less indicates the presence of considerable amount of boron in ASZ. Figure 5 shows the presence of boron in phases A and B. This liquid transforms to phases A and B during cooling from the bonding temperature. Boron tends more to

### Table 3. EDS point analysis of joint band phases, A, B, and C (wt%)

<table>
<thead>
<tr>
<th>Element</th>
<th>Phase</th>
<th>A</th>
<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td></td>
<td>0.3</td>
<td>0.4</td>
<td>0.4</td>
</tr>
<tr>
<td>Mn</td>
<td></td>
<td>0.6</td>
<td>1.1</td>
<td>0.7</td>
</tr>
<tr>
<td>Fe</td>
<td></td>
<td>70.3</td>
<td>78.6</td>
<td>85.2</td>
</tr>
<tr>
<td>Ni</td>
<td></td>
<td>7.2</td>
<td>9.3</td>
<td>10.4</td>
</tr>
<tr>
<td>Mo</td>
<td></td>
<td>21.6</td>
<td>10.6</td>
<td>3.3</td>
</tr>
</tbody>
</table>

Fig. 5. EDS point analysis of phases A, B and C in joint made at 1473 K for 30 min.

Fig. 6. Fe-Ni-Mo ternary diagram isothermal section at 1473 K [19].
combine with Mo and Fe rather than with Ni [20]. According to Fe-B and Mo-B binary phase diagrams, the presence of a low amount of boron in Fe and Mo leads to the formation of Fe₂B and Mo₂B at the lower temperature of 1473 K respectively [19]. Therefore, phases A and B are Mo₂B (borides of Fe and Mo) and γ respectively at temperature less than 1473 K.

As mentioned previously, at the bonding temperature of 1473 K, the time of complete isothermal solidification was 40 minutes (Fig. 3d). Therefore to produce dual phase steels with different martensite morphology (FCM, FFM, and FBM microstructures) in the base metal, joints were made according to Table 2 heat treatment cycles. Microstructure of homogenized joints is shown in Fig. 7. As can be seen, each microstructure consists of three distinct zones, joint band (JB) or ISZ, diffusion affected zone (DAZ) and base metal (BM). Microstructure of base metal far from the joints is similar to those in Fig. 2.

Table 4 shows EDS point analysis results of joints band prepared according to Table 2 heat treatment cycles (Fig. 7). As can be seen, homogenization heat treatment caused redistribution of alloying elements between joint band and base metal. But the alloying elements concentration depends on the holding time at the homogenization heat treatment temperature.

According to Fe-Ni binary diagram [19] and Table 4, at the homogenization temperature of 1008 K the joint band is a solid solution of Ni in γFe which transforms to martensite by quenching from 1008 K to room temperature (Fig. 8).

Base metal microstructure is ferrite and martensite, but the morphology of martensite depends on used heat treatment cycle according to Table 2. That is, ferrite - continuous martensite (FCM) Fig. 7(a) (or Fig. 2(a)), ferrite - fibrous martensite (FFM) Fig. 7(b) (or Fig. 2(b)), and ferrite - blocky martensite (FBM) Fig. 7(c) (Fig. 2(c)). The DAZ microstructure is mainly ferrite and martensite, volume fraction of martensite depends on holding time at 1008 K. As can be seen from Fig. 7, martensite volume fraction in DAZ of FBM microstructure (Fig. 7(c)) is less than the DAZ martensite of the other two microstructures. This can be due to the more diffusion of carbon from DAZ of this joint because of higher homogenization time (80 minutes).

3.2. Mechanical properties

3.2.1. Hardness

Microhardness profile from the joint band center of homogenized joint toward the base metal is plotted in Fig. 9. Each data point in Figure is average of 5 measurements. As can be seen, the hardness variation is the same for dual phase steels with different martensite morphology. Joint band hardness is more than DAZ and base metal because of its fully martensitic microstructure (Fig. 8) in comparison to DAZ and base metal microstructures which are ferrite and martensite (Fig. 7 or 2). The DAZ hardness is less than the base metal due to the less volume fraction of martensite in this zone.

3.2.2. Shear strength

Shear tests on base metal samples, with microstructure shown in Fig. 2, and bonded samples, at optimum bonding time and then homogenized to produce dual phase steels with
different martensite morphology (Fig. 7) is given in Table 5. As can be seen, dual phase steel (BM) with ferrite - fibrous martensite (FFM) has the highest shear strength. This is because of the finer ferrite grains and more uniform distribution of martensite in FFM dual phase steel in comparison to the two other microstructures [10,12,21,22]. The joint shear strength of this steel is also higher than those of the other joints. Table 5 also shows the joint to base metal shear strengths ($\tau_J/\tau_{BM}$) and indicates that the joint shear strength in FCM and FFM dual phase steels are about or greater than 85% that of the base metal with the same microstructure. In joint of steel with FBM microstructure the joint shear strength is about 69% that of the base metal with this microstructure. As will discuss in the next section, fracture occurred from DAZ and DAZ of this bonded sample has less martensite volume fraction in comparison to the other two bonded samples DAZ.

3.2.3. Fracture surface

Figure 10 shows the fracture path in joint of steel with FBM microstructure. As can be seen, fracture occurred through the DAZ. Fracture of joints in steels with FCM and FFM microstructures is also happened through the same zone. This is due to the less martensite volume fraction in the DAZ as shown

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**Table 5.** Average shear strength of base metal and joints made at the optimum time and homogenized to produce for three types of DP steel microstructure

<table>
<thead>
<tr>
<th>Microstructure</th>
<th>FCM (MPa)</th>
<th>FFM (MPa)</th>
<th>FBM (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shear Strength Joint</td>
<td>530 ± 15</td>
<td>598 ± 16</td>
<td>387 ± 13</td>
</tr>
<tr>
<td>Base Metal</td>
<td>622 ± 12</td>
<td>657 ± 8</td>
<td>557 ± 18</td>
</tr>
<tr>
<td>$\tau_J/\tau_{BM}$ (%)</td>
<td>85</td>
<td>91</td>
<td>69</td>
</tr>
</tbody>
</table>

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**Fig. 9.** Microhardness variation from the joint center toward the base metal.

**Fig. 10.** Failure path in joint region of bonded sample with FBM microstructure.

**Fig. 11.** Fracture surfaces for FCM, (a) BM, (b) bonded sample.
in Fig. 7.
Figure 11 shows photograph of sheared surfaces of bonded sample and base metal for steel with FCM microstructure. Presence of elongated dimples on the fracture surface indicates ductile fracture for both samples [23]. Dimples on the fracture surface of bonded sample are greater than those in the base metal. As mentioned, the fracture pass was from the DAZ which has less volume fraction of martensite. This can be seen by comparison of Fig. 2(a) (base metal) with DAZ of Fig. 7(a). Fracture surfaces of sheared specimens of bonded and base metal for the other two microstructure, FFM and FBM microstructures, were the same.

4. CONCLUSION

Transient liquid phase bonding of a carbon steel carried out at 1473 K for different holding time, using a Fe based interlayer, to produce dual phase steels with different martensite morphology during bonding process. From the results:
(1) At the bonding time of 10, 20, 30 minutes, there was two distinct zones, ASZ and ISZ, in the joint band. But with increasing the bonding time from 10 minutes, the ASZ decreased and completely disappeared at the bonding time of 40 minutes.
(2) The ISZ was a Ni solid solution of γFe at the bonding and homogenization temperatures. This solid solution transformed to martensite during quenching from the bond homogenization temperature. The ASZ was borides of Fe and Mo.
(3) Dual phase steels with different martensite morphology, FCM, FFM, and FBM, were produced successfully during TLP bonding heat treatment cycle.
(4) Shear strength of bonded samples was about or more than 85% that of the base metal with FCM and FFM microstructure. But for microstructure of FBM it was about 70%.
(5) Shear fracture occurred from DAZ of all bonded samples with ductile manner. The base metal fracture, with the same microstructure as the bonded samples, occurred also in ductile mode.

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