Effect of Cr and C on microstructure evolution of medium carbon steels during friction stir welding process and their mechanical property

C Cheng 1, K Kadoi 1, S Tokita 1, H Fujii 1, K Ushioda 1 and H Inoue 1

1 Joining and Welding Research Institute, Osaka university, Osaka, Japan

Email: kadoi@jwri.osaka-u.ac.jp

Abstract. The aim of the present study was to investigate the effect of chromium and carbon in medium carbon steels on the microstructure evolution during FSW process and their mechanical property. The materials are dynamically recrystallized in austenite during FSW followed by phase transformation from austenite to ferrite, pearlite, bainite, and martensite. In the case of 0.2%C, the microstructure of the stir zone transited from pearlite and ferrite to bainite and martensite with the increase in chromium content. On the other hand, in the case of 0.4%C, the microstructure transited from pearlite to martensite with the increase in chromium content. The specimens of 0.2%C-4%Cr, 0.4%C-2%Cr and 0.4%C-4%Cr showed high tensile strength of over 1.5 GPa. In addition, even though the microstructures in the stir zones of the specimens are martensite, the elongation of 0.2%C-4%Cr was 31% while those of the others were less than 5%. TEM observation revealed that many tiny carbides precipitated in martensite of the stir zone of 0.2%C-4%Cr and micro twin formed in martensite in those of 0.4%C-2%Cr and 0.4%C-4%Cr. Thus, it was considered that auto-tempering occurred in the stir zone of 0.2%C-4%Cr during FSW process. The carbide precipitation in martensite is more effective to improve the tensile elongation compared with micro twin formation.

1. Introduction

Medium carbon steels have advantages of high strength with low cost. Therefore, they have a wide range of applications in industries. However, the weldability and the mechanical properties of the fusion-welded joint of the steels deteriorate with increasing in C content due to the formation of hardened phase such as martensite. Martensite with high carbon content is brittle and susceptible to cracking. Furthermore, the ductility of carbon steel also decreases due to the formation of brittle martensite during welding process [1].

It is known that chemical composition (alloying elements) and thermal cycle during welding corresponding to weld heat input mainly influence on the microstructure evolution and the morphology. In the case of the chemical composition, the alloying elements owing high affinity to carbon affect the phase transformation from austenite during welding. Chromium is one of the candidate elements since chromium enhances high hardenability and an attractive interaction with carbon (formation of chromium carbide) [2]. The ductility of martensite should also modify by the formation of the carbide [3,4]. On the other hand, in the case of the thermal cycle during welding, it is required to apply the proper welding process and to control the heat input and the peak temperature. Friction stir welding (FSW) process is a solid-state joining process with low peak temperature and low heat input [5]. Thus, the application of FSW process on medium carbon steels induces the modification of microstructure such as martensite.
phase compared to the fusion welding process. Besides, FSW process has also an advantage of refining austenite grains by dynamic recrystallization corresponding to work strain.

Even though there are considerable numbers of study on the application of FSW process to carbon steels, the influence of alloying elements in medium carbon steels on microstructural evolution during FSW is still under the discussion due to complicate phenomena such as the dynamic recrystallization of austenite and the phase transformation \cite{6-8}. The aim of this study is to investigate the effect of chromium and carbon in medium carbon steels on the microstructure evolution during FSW process and their mechanical properties (tensile strength and hardness) and the microstructure.

2. Experimental method

Table 1 shows chemical compositions of specimens. Carbon and chromium contents were varied as 0.2 and 0.4 mass% and 2 and 4 mass%, respectively. The specimens were fabricated through vacuum induction melting under argon atmosphere and hot rolling. The specimen surface was machined after the hot rolling. The specimen geometry for FSW test was 180 - 200W × 45L × 3.3t mm. The chemical compositions were measured with an optical emission spectrometer. Hereafter, the base composition of Fe-0.2%C-2%Cr is called 0.2C-2Cr for simplicity. The other specimens are also called 0.2C, 0.2C-4Cr, 0.4C, 0.4C-2Cr, and 0.4C-4Cr, respectively.

Stir in plate FSW were carried out on the base metal plates by using a loading controlled FSW machine. The tool was made of WC and the geometry was the shoulder diameter of 15 mm, the probe diameter of 6 mm, and the probe length of 3.1 mm as depicted in figure 1. FSW was performed under welding speed of 400 rpm and the rotation speed was 150 mm/min. The load was adjusted according to the base metal. The tool was tilted 3 degrees. The peak temperature during FSW under the conditions is considered above Ac3 temperature in order to investigate the phase transformation after fully austenitizing \cite{6}.

The cross-sectional microstructures of the specimens were analyzed through an optical microscope (OM), a field emission type scanning electron microscope (SEM, JEOL JSM-7001FA) and a transmission electron microscope (TEM, JEOL 2100F). The specimens were prepared for OM and SEM observations by chemical etching with 3% nital solution after final polishing with colloidal silica. The prior austenite grain size in the stir zone was measured by liner intercept method using the specimen etched by 1.5% picric acid. The specimens for TEM observation were prepared thinning by mechanical polishing and further thinned by electropolishing in a twin-jet polisher (Struers TenuPol-5).

| Table 1 Chemical compositions of base metal (mass\%) |
|-----------------|-----------|-------|-------|-------|-------|-------|-------|
|                 | C         | Cr    | Si    | P     | S     | Mn    |
| 0.2C            | 0.16      | 0.0   | 0.010 | <0.01 | <0.01 | 0.01  |
| 0.2C-2Cr        | 0.19      | 1.97  | 0.014 | <0.01 | <0.01 | <0.01 |
| 0.2C-4Cr        | 0.20      | 4.01  | 0.017 | <0.01 | <0.01 | <0.01 |
| 0.4C            | 0.39      | 0.0   | 0.010 | <0.01 | <0.01 | <0.01 |
| 0.4C-2Cr        | 0.39      | 1.97  | 0.014 | <0.01 | <0.01 | <0.01 |
| 0.4C-4Cr        | 0.39      | 4.02  | 0.017 | <0.01 | <0.01 | <0.01 |

Fig. 1 Schematic of FSW tool. Fig. 2 Schematic of specimen geometry for tensile test
The Vickers hardness and the tensile property in the stir zones were determined as the mechanical property. The small special test specimen for tensile test was paralleled length of 3 mm and width of 2 mm as shown in figure 2. The entire paralleled area was covered with the stir zone to investigate the tensile property of the stir zone. The tensile test was carried out under 0.04 mm/min of a cross-head speed at room temperature. The strain was measured by tracing gauge marks (Vickers indents) on the specimen using a high-speed camera.

3. Results and discussion

3.1. Microstructure of stir zone

Figure 3 shows SEM images of microstructure in the stir zone of the specimens with different carbon and chromium contents. In the case of 0.2 % C, the microstructure of the stir zone changed from pearlite and ferrite to bainite and martensite with the increase in chromium content. In contrast, in the case of the carbon content of 0.4%, the microstructures are pearlite in 0.4C and martensite in both 0.4C-2Cr and 0.4C-4Cr. The martensite found in the specimens of 0.2C-0.4Cr, 0.4C-2Cr and 0.4C-4Cr is mainly lath martensite. However, the detailed morphologies are different.

Figure 4 represents prior austenite grains in stir zones of each specimen. The stir zones in the specimens containing chromium consist of fine grains which is around 20 µm. The average grain sizes
of the prior austenite in the stir zones of 0.2C-2Cr, 0.2C-4Cr, 0.4C-2Cr, and 0.4C-4Cr are 25.5, 21.8, 24.1, and 19.4 μm, respectively. It was impossible to determine the prior austenite grain size in 0.2C and 0.4C specimens (no chromium containing) because the grain boundaries were not clear. The prior austenite grains tend to be refined with the increase of chromium content regardless of carbon content. Addition of carbon also induces grain refining. Thus, the recrystallization of austenite by intense plastic deformation and heat generation during FSW attributes to refine the grains.

In order to analyze the detailed morphology of the martensite which was observed in figure 3, TEM-EDS analysis was carried out on the specimens of 0.2C-4Cr, 0.4C-2Cr and 0.4C-4Cr. Figure 5 shows TEM images of the stir zone of the specimens. Tiny precipitates (< 100 μm) distribute in the martensite laths in 0.2C-4Cr. The dislocations are also observed around the precipitates. Dense micro twins are mainly observed in 0.4C-2Cr and 0.4C-4Cr. Additionally, there is little precipitation in spite of high carbon content compared to 0.2C-4Cr. TEM-EDS spectroscopy maps in the stir zone of 0.2C-4Cr and 0.4C-4Cr are shown in figure 6. Carbon and iron are detected at the tiny precipitates in 0.2C-4Cr. Thus, the precipitates must be identified as iron carbide. There is no partitioning of chromium because the diffusion of chromium atoms after martensitic transformation is difficult under high cooling rate by FSW. On the other hand, in the case of 0.4C-4Cr, the distributions of carbon and chromium are uniform. This is caused by relatively low martensitic start temperature (Ms) as compared with that in 0.2C-4Cr. Thus, carbide is hardly precipitated in the stir zone of 0.4C-4Cr.

The stir zones of the specimens were firstly austenitized since the peak temperature was considered to be above Ac3 temperature during FSW process and then transformed into ferrite, pearlite, bainite and martensite depending on chemical compositions and cooling rate. Besides, chromium is known to induce a separation of CCT curve into ferrite formation field and bainite formation field and simultaneously retards the kinetics of phase transformation. Carbon is austenite stabilizing element, the incubation period of austenite to transform to pearlite and bainite increases with the increase of carbon content [9]. Thus, pearlite and ferrite were transformed from austenite in 0.2C and 0.4C without chromium. Bainite transformation also occurred in 0.2C-2Cr during the FSW process. The further increase in chromium content delays the bainite transformation [10]. Thus, martensite formed in 0.2C-4Cr, 0.4C-2Cr and 0.4C-4Cr since the super-cooled austenite in the stir zone of 0.4C with chromium transform to martensite at further lower Ms temperature compared to that of 0.2C-4Cr. The carbide precipitation in 0.2C-4Cr is considered by the auto-temper process after martensitic transformation since Ms temperature is relatively high. In order to investigate the carbide precipitation in auto-tempered martensite, the heat treatment at 400 °C for 1 hour was applied for the specimen of 0.4C-4Cr. The TEM image and TEM-EDS mappings are shown in figure 7. The carbide precipitations were a size of 200 - 300 nm. The precipitates are rich in carbon, chromium and iron. Moreover, it should be noticed that micro-twins in 0.4C-4Cr still remained even after the heat treatment.
3.2. Mechanical properties
The hardness of the stir zones of 0.2C–2Cr, 0.2C–4Cr, 0.4C–2Cr, and 0.4C–4Cr are 148, 365, 484, 266, 669, and 707 HV, respectively. In both cases of the carbon content of 0.2C and 0.4C, the hardness increases with the chromium content. This tendency corresponds to the microstructural transition with carbon and chromium contents as shown in figure 3. Even though the microstructures of stir zone in 0.2C-4Cr, 0.4C-2Cr, and 0.4C-4Cr are commonly martensite, the hardness of 0.2C-4Cr is the much lower than those of the other martensite in 0.4C-2Cr and 0.4C-4Cr. This should be caused by auto-tempering and hardness of martensite owing to carbon content.

Figure 8 shows the stress-strain curves of the specimens with different carbon and chromium contents. The ultimate strength of the stir zone in 0.4C, 0.2C-4Cr, 0.4C-2Cr and 0.4C-4Cr are 852 MPa, 1601 MPa, 2181 MPa and 2479 MPa, respectively. The total elongation of the stir zone in 0.4C, 0.2C-4Cr, 0.4C-2Cr and 0.4C-4Cr are 35.1%, 29.6%, 4.3% and 4.1%, respectively. It is remarkable that material still keeps high ductility with high strength, although martensite formed in 0.2C-4Cr. On the other hand, 0.4C-2Cr and 0.4C-4Cr showed very low elongation and brittle fracture.

The strength and the elongation of tempered 0.4C-4Cr are 1715 MPa and 19.1%. The tempering heat treatment could modify the elongation from 4.2 to 19.1% and the strength keeps high. Besides, the strength and the elongation of tempered 0.4C-4Cr are close to those of the 0.2C-4Cr auto-tempered martensite. It is consistent with that martensite is softened after tempering in 0.2C-4Cr. The carbide precipitation observed in the stir zone of 0.2C-4Cr indicated that auto-temper process occurred during the FSW process. In contrast, high carbon content attributes to the ultrahigh ultimate strength and low elongation by the formation of martensite with micro twins. It is worth noting that auto-tempered lath-martensitic structure of 0.2C-4Cr exhibited an excellent balance of strength and ductility among the steels with the commonly martensitic structure in the stir zone of 0.2C-4Cr, 0.4C-2Cr and 0.4C-4Cr.
4. Summary
The microstructure of the stir zone of the specimens containing 0.2 % C, changed from pearlite and ferrite to bainite and martensite with the increasing chromium content. In the case of the carbon content of 0.4%, the microstructures are pearlite in 0.4C and martensite in both 0.4C-2Cr and 0.4C-4Cr. The martensite found in the specimens of 0.2C-0.4Cr, 0.4C-2Cr and 0.4C-4Cr is mainly lath martensite.

With the increase of chromium and carbon contents, the strength of the stir zone increased and the elongation decreased. The specimens of 0.2%C-4%Cr, 0.4%C-2%Cr and 0.4%C-4%Cr showed high tensile strength of over 1.5 GPa. In addition, even though the microstructures in the stir zones of the specimens are martensite, the elongation of 0.2%C-4%Cr was 31% while those of the others were less than 5%. The auto tempering process induced to tiny carbide formation within lath martensite in 0.2C-4Cr specimen. Micro twins mainly formed in 0.4C-2Cr and 0.4C-4Cr specimens. Thus, the tiny carbide precipitation in the martensite has a significant effect on the balance between high strength and good elongation.

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