CHAPTER 6

METALLURGICAL CHARACTERIZATION OF
FRICION STIR WELDED AA6061/ZrB<sub>2</sub> MMC

6.1 INTRODUCTION

FSW produces significant metallurgical changes along the weld line. It is essential to study the microstructural changes which influence the mechanical behavior of the joint. This chapter presents the metallurgical characterization of friction stir welded AA6061/ZrB<sub>2</sub> MMC. The effect of content of ZrB<sub>2</sub> particulates on microstructure and microhardness is analyzed. The fracture surface and worn surface of selected specimens are also discussed. The mode of fracture and wear are identified. The post weld heat treatment of optimized weldment is also presented.

6.2 METALLURGICAL STUDIES

6.2.1 Macrostructure

Five specimens were prepared perpendicular to the joint line from the welded plates which had different content of ZrB<sub>2</sub> particles (T01, T15, T23, T24 and T29). All the specimens were polished using standard metallographic technique (Initial polishing with different grades of emery sheets and fine polishing with diamond paste up to 2 μ size) and etched with a color etchant containing 4 g potassium permanganate, 1 g sodium hydroxide in 100 ml distilled water. The digital image of the macrostructure of the etched specimens was captured using a digital optical scanner.

6.2.2 Microstructural Analysis
The etched specimens were observed using an optical microscope (OLYMPUS-BX51M) and a scanning electron microscope (JEOL-JSM-6390). Photo micrographs were taken on various zones.

6.2.3 Microhardness Survey

The microhardness was measured using a microhardness tester (MITUTOYO-MVK-H1) at 500 g load applied for 15 seconds along the cross section of specimen obtained perpendicular to the welding direction. The indentation was made up to 15 mm (one indentation/mm) either side of the centre of weld line at 1 mm from top, middle and 1mm above from the bottom of specimens.

6.2.4 Fracture and Worn surface Morphology

The fracture surfaces (T07, T14, T21, and T27) and worn surfaces (T02, T11, T23, T24 and T30) of selected specimens were observed using a scanning electron microscope.

6.2.5 Post Weld Heat Treatment (PWHT)

The plates welded using optimized parameters OP1 was solution treated at 520°C for 1 h followed by artificial ageing at 165°C for 18 h (Priya et al 2009). The microstructure was observed using a scanning electron microscope and microhardness was measured as given in section 6.2.3.

6.3 RESULTS AND DISCUSSIONS

6.3.1 Macrostructure

Figure 6.1 shows the macrostructure of friction stir welded AA6061/10wt. % ZrB₂ MMC. The different zones typically present in FSW of aluminum alloys are visible. The macrostructure consists of parent composite (BM), heat affected zone (HAZ), thermomechanically affected zone (TMAZ) and weld zone (WZ). Similar zones are observed in other welded joints. TMAZ and HAZ constitute the transition zone. The details of those zones are explained in the following section. The little
sensitivity of the composite to thermal variations has limited the transitional zone to TMAZ.

![Figure 6.1 Macrostructure of FS Welded AA6061/10wt.% ZrB₂ MMC (T24)](image)

**Figure 6.1 Macrostructure of FS Welded AA6061/10wt.% ZrB₂ MMC (T24)**

### 6.3.2 Microstructural Analysis

Figures 6.2-6.6 depict the microstructure of parent composite of friction stir welded AA6061/ZrB₂ MMCs having different amount of ZrB₂ particles. Those microstructure details are already described in section 3.4.2, chapter 3. The etchant used reveals ZrB₂ particles in white color, Si phase in blue color and Mg₂Si phase in black color.

Figures 6.6-6.11 depict the microstructure of HAZ of friction stir welded AA6061/ZrB₂ MMCs having different amount of ZrB₂ particles. The microstructure of HAZ and parent composite are almost identical except in the matrix alloy. The dendritic structure in the matrix alloy is slightly refined by frictional heat subsequent to welding. Some amount of Mg₂Si phase is dissolved and the spacing between dendritic arms is reduced. The grain size and distribution of ZrB₂ particles in HAZ of welded composites does not show appreciable difference compared to parent composites. Amirizad et al (2006) attributed this occurrence to the little thermal sensitivity of MMCs. The ceramic particles have lower thermal conductivity compared to matrix alloy which act as thermal barriers. Hence, the transition zone is limited to TMAZ in friction stir welded MMCs.
Figure 6.2 Photomicrograph of BM of FS Welded AA6061 (T23)

Figure 6.3 Photomicrograph of BM of FS Welded AA6061/2.5wt.% ZrB$_2$ MMC (T01)
Figure 6.4 Photomicrograph of BM of FS Welded AA6061/5wt.% ZrB$_2$ MMC (T29)

Figure 6.5 Photomicrograph of BM of FS Welded AA6061/7.5wt.% ZrB$_2$ MMC (T15)
Figure 6.6 Photomicrograph of BM of FS Welded AA6061/10wt.% ZrB₂ MMC (T24)

Figure 6.7 Photomicrograph of HAZ of FS Welded AA6061 (T23)
Figure 6.8 Photomicrograph of HAZ of FS Welded AA6061/2.5wt.% ZrB$_2$ MMC (T01)

Figure 6.9 Photomicrograph of HAZ of FS Welded AA6061/5wt.% ZrB$_2$ MMC (T29)
Figure 6.10 Photomicrograph of HAZ of FS Welded AA6061/7.5wt.% ZrB$_2$ MMC (T15)

Figure 6.11 Photomicrograph of HAZ of FS Welded AA6061/10wt.% ZrB$_2$ MMC (T24)
Figures 6.12-6.16 depict the microstructure of transition zone of friction stir welded AA6061/ZrB$_2$ MMCs having different amount of ZrB$_2$ particles. The boundary between the weld zone and TMAZ are visible quite clearly. TMAZ is plastically deformed and thermally affected. TMAZ exhibits highly elongated grains of aluminum alloy without recrystallized microstructure. The elongated grains of the matrix alloy in TMAZ have a rotation up to 90°. TMAZ reveals the alignment of ZrB$_2$ particles in the vertical direction. A parallel band like distribution of particles is observed. Frictional heat generated by the rotating tool and application of stresses induce plastic deformation in TMAZ and stretches ZrB$_2$ particles along the shear stress directions. TMAZ on both the sides (advancing and retreating) exhibits similar structure. It is interesting to note that the width of transition zone reduces with increased content of ZrB$_2$ particles. The width of transition zone is about 200 µm and 50 µm respectively in friction stir welded AA6061 and AA6061/10wt.% ZrB$_2$ MMC.

Figure 6.12 Photomicrograph of Transition Zone of FS Welded AA6061 (T23)
Figure 6.13 Photomicrograph of Transition Zone of FS Welded AA6061/2.5wt.% ZrB₂ MMC (T01)

Figure 6.14 Photomicrograph of Transition Zone of FS Welded AA6061/5wt.% ZrB₂ MMC (T29)
Figure 6.15 Photomicrograph of Transition Zone of FS Welded AA6061/7.5wt.% ZrB$_2$ MMC (T15)

Figure 6.16 Photomicrograph of Transition Zone of FS Welded AA6061/10wt.% ZrB$_2$ MMC (T24)
Figures 6.17-6.21 depict the microstructure of weld zone of friction stir welded AA6061/ZrB2 MMC having different amount of ZrB2 particles. The dendrite structure of the matrix alloy is entirely altered in friction stir welded AA6061. Weld zone shows fine-equiaxed recrystallized grains due to heavy plastic deformation followed by dynamic recrystallization. Typical fusion welding defects such as porosity and segregation are not seen. The weld zone displays a homogeneous distribution of ZrB2 particles irrespective of its content. The applied frictional heat did not promote any reaction between the matrix and ZrB2 particles. High plastic strain caused by stirring redistributes ZrB2 particles. The presence of the reinforcement particles increase the nucleation sites (Ceschini et al 2007a) which leads to the reduction of grain size of recrystallized aluminum matrix. The number of particles in the weld zone appears to be larger compared to parent composites. The stirring action of the tool fragments several clusters. Partially broken clusters are also visible in the weld zone. It is interesting to note that most of the fragmented particles retain the spherical shape. This is due to the abrading action of the tool which blends the sharp edges of the fragments and tends to round them.

Figure 6.17 Photomicrograph of Weld Zone of FS Welded AA6061 (T23)
Figure 6.18 Photomicrograph of Weld Zone of FS Welded AA6061/2.5wt.% ZrB$_2$ MMC (T01)

Figure 6.19 Photomicrograph of Weld Zone of FS Welded AA6061/5wt.% ZrB$_2$ MMC (T29)
Figure 6.20 Photomicrograph of Weld Zone of FS Welded AA6061/7.5wt.% ZrB$_2$ MMC (T15)

Figure 6.21 Photomicrograph of Weld Zone of FS Welded AA6061/10wt.% ZrB$_2$ MMC (T24)
Figures 6.22-6.26 depict the SEM microstructure of weld zone of friction stir welded AA6061/ZrB$_2$ MMCs having different amount of ZrB$_2$ particles. Figure 6.22 is the weld zone microstructure of friction stir welded matrix alloy AA6061. The fine recrystallized grains along with second phase particles of the alloy are clearly seen. The average grain size is 8 $\mu$m. The grains are not seen in the weld zone microstructure of friction stir welded AA6061/ZrB$_2$ MMCs. The fragmentation of ZrB$_2$ clusters is clearly seen. Each cluster present in the parent composite as shown in Figures 3.20-3.23 provided in chapter 3 is thoroughly broken into fine particles of different sizes. The average particle size in the weld zone is approximately 2-3 $\mu$m. FSW results in the redistribution of inter granular particles into intra granular particles.

![SEM Photomicrograph of Weld Zone of FS Welded AA6061 (T23)](image_url)

**Figure 6.22 SEM Photomicrograph of Weld Zone of FS Welded AA6061 (T23)**
Figure 6.23 SEM Photomicrograph of Weld Zone of FS Welded AA6061/2.5wt.% ZrB₂ MMC (T01)

Figure 6.24 SEM Photomicrograph of Weld Zone of FS Welded AA6061/5wt.% ZrB₂ MMC (T29)
Figure 6.25 SEM Photomicrograph of Weld Zone of FS Welded AA6061/7.5wt.% ZrB$_2$ MMC (T15)

Figure 6.26 SEM Photomicrograph of Weld Zone of FS Welded AA6061/10wt.% ZrB$_2$ MMC (T24)
6.3.3 Microhardness Survey

Figures 6.27-6.31 show the microhardness profiles obtained across friction stir welded AA6061/ZrB\textsubscript{2} MMC having different amount of ZrB\textsubscript{2} particles. It resembles a typical “Bell” shape. The hardness of the weld zone is higher than that of the parent composite in all the joints. The hardness of the transition zone varies between those of parent composite and weld zone. The hardness variation from top to bottom of the joints was insignificant. The maximum hardness recorded in the welded joints increased with increased content of ZrB\textsubscript{2} particles.

Weld zone hardening is described as follows. FSW closes the presence of micro porosities in the cast composite. The grain size of aluminum in the weld zone is reduced by dynamic recrystallization. The size of the ZrB\textsubscript{2} particles reduces towards weld zone and the number of particles is increasing. The weld zone is filled with more particles homogeneously dispersed compared to parent composite. This contributes to increase in dislocation density in the weld zone. The age hardening and softening characteristics of cast composite is different to that of wrought/heat treated composite. The net result is hardening of weld zone.

![Microhardness Profile](image)

Figure 6.27 Hardness Profile across FS Welded AA6061 (T23)
Figure 6.28 Hardness Profile across FS Welded AA6061/2.5wt.% ZrB₂ MMC (T01)

Figure 6.29 Hardness Profile across FS Welded AA6061/5wt.% ZrB₂ MMC (T29)
Figure 6.30 Hardness Profile across FS Welded AA6061/7.5wt.% ZrB$_2$ MMC (T15)

Figure 6.31 Hardness Profile across FS Welded AA6061/10wt.% ZrB$_2$ MMC (T24)
6.3.4 Fracture Surface Morphology

Figures 6.32-6.35 depict the SEM micrographs of fracture surface of friction stir welded AA6061/ZrB₂ MMCs having different amount of ZrB₂ particles. The fracture surfaces of welded composites are relatively flat compared to the fracture surfaces of parent composites as shown in Figures 3.29-3.30, chapter 3 which indicates a loss of ductility after welding. The degree of flatness of fracture surfaces increases as the amount of ZrB₂ particles is increased. The fracture surface of friction stir welded AA6061/2.5wt.% ZrB₂ MMC shows a net work of fine dimples. The dimple size is not uniform in the fracture surface of friction stir welded AA6061/5wt.% ZrB₂ MMC due to fragmentation of ZrB₂ particles and partially broken clusters. The fracture surfaces of friction stir welded AA6061/7.5 and 10wt.% ZrB₂ MMCs are absolutely flat which indicates brittle behavior of the joint. The typical failure mechanism of friction stir welded MMCs may be as follows: (i) breaking of large size particles, (ii) detachment of particles at matrix-particle interface which leads to nucleation of voids and (iii) growth and coalescence of voids.

![SEM Micrograph of Fracture Surface of FS Welded AA6061/2.5wt.% ZrB₂ MMC (T07)](image)

Figure 6.32 SEM Micrograph of Fracture Surface of FS Welded AA6061/2.5wt.% ZrB₂ MMC (T07)
Figure 6.33 SEM Micrograph of Fracture Surface of FS Welded AA6061/5wt.% ZrB$_2$ MMC (T27)

Figure 6.34 SEM Micrograph of Fracture Surface of FS Welded AA6061/7.5wt.% ZrB$_2$ MMC (T14)
6.3.5 Worn surface Morphology

Figures 6.36-6.40 depict the SEM micrographs of worn surface of friction stir welded AA6061/ZrB$_2$ MMC having different amount of ZrB$_2$ particles. A change in wear mode is observed with increase in content of ZrB$_2$ particles which can be attributed to the homogeneous distribution of ZrB$_2$ particles in the weld zone as a result of FSW. The wear mode changes from adhesion (Figures 6.36 and 6.37) to abrasive wear (Figures 6.38- 6.40). Friction stir welded matrix alloy and composite containing 2.5 wt.% of ZrB$_2$ particles exhibit adhesion wear mode which shows large amount of plastic flow of material. The frictional heat increases the worn surface temperature which causes plastic deformation and dislocation in the inner surface of the composites. The congestion of dislocation results in stress concentration and initiation of cracks. Friction stir welded composites containing ZrB$_2$ particles above 2.5 wt.% exhibit abrasive wear mode. The width of the wear track increases as the amount of ZrB$_2$ particles is increased. The abrasive wear is the
result of ZrB$_2$ particles on the worn surface and the abrasive dusts between two surfaces. ZrB$_2$ particles bear the load initially. As sliding wear proceeds, the frictional heat softens the surface layer. The difference in thermal expansion coefficient between the matrix and the ZrB$_2$ particles creates an interface stress. When the interface stress exceeds the bond strength the particles are pulled off. The pulled off particles begin to act as wear particles during the rest of sliding wear.

### 6.2.6 Post Weld Heat Treatment

Figures 6.41 and 6.42 respectively show the SEM photomicrographs of FS welded AA6061/10wt.% ZrB$_2$ MMC at optimized parameters OP1 before and after PWHT. Coarsening of grains is observed. The XRD pattern of the same composite (Figure 6.43) shows an increase in Mg$_2$Si peaks after PWHT which indicates the formation of fine precipitates. The solutionizing treatment results in dissolution of precipitates throughout the weldment. Rapid cooling following solutionizing creates supersaturated state with large number of quenched in vacancies throughout the weldment. Ageing following quenching results in homogeneous precipitation of strengthening phase throughout the weldment which increases the hardness throughout the weldment (Figure 6.44).

![Figure 6.36 SEM Micrograph of Worn surface of FS Welded AA6061 (T23)](image-url)
Figure 6.37 SEM Micrograph of Worn surface of FS Welded AA6061/2.5wt.% ZrB$_2$ MMC (T02)

Figure 6.38 SEM Micrograph of Worn surface of FS Welded AA6061/5wt.% ZrB$_2$ MMC (T30)
Figure 6.39 SEM Micrograph of Worn surface of FS Welded AA6061/7.5wt.% ZrB$_2$ MMC (T11)

Figure 6.40 SEM Micrograph of Worn surface of FS Welded AA6061/10wt.% ZrB$_2$ MMC (T24)
Figure 6.41 SEM Photomicrograph of Weld Zone of FS Welded AA6061/10wt.% ZrB$_2$ MMC at Optimized Parameters OP1 before PWHT

Figure 6.42 SEM Photomicrograph of Weld Zone of FS Welded AA6061/10wt.% ZrB$_2$ MMC at Optimized Parameters OP1 after PWHT
Figure 6.43 XRD Results of FS Welded AA6061/10wt.% ZrB₂ MMC at Optimized Parameters OP1

Figure 6.44 Hardness Profile across FS Welded AA6061/10wt.% ZrB₂ MMC at Optimized Parameters OP1
6.4 **SUMMARY**

i. Friction stir welded AA6061/ZrB$_2$ MMC joint exhibits the presence of different zones such as weld zone, thermo mechanically affected zone and heat affected zone.

ii. The transition zone is limited to TMAZ. The width of transition zone reduced with increased content of ZrB$_2$ particles.

iii. The weld zone is characterized with a homogeneous distribution of ZrB$_2$ particles.

iv. ZrB$_2$ particles are fragmented subsequent to FSW due to the stirring action of the tool.

v. FSW increases the hardness of weld zone than that of the parent composite due to particle fragmentation.

vi. The welded joints show reduced ductile behavior and the wear mode changes from adhesive to abrasive with increased content of ZrB$_2$ particles.

vii. PWHT improved the hardness across the welded joint and enhanced the joint strength.