国士舘大学審査学位論文

「Creation of Innovative Functions for Zn-22Al Superplastic Alloy through Friction Stir Processing」

(摩擦攪拌プロセスによる Zn-22Al 超塑性合金の革新的機能の創成)

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Abstract

This dissertation is a systematic study on the developments of innovative functions for Zn-22Al superplastic alloy through friction stir processing (FSP), which is a type of friction stir welding (FSW).

Superplastic materials are fine-grained polycrystalline solids that have recently gathered interest because of their excellent tensile elongation and behavior in superplastic forming (SPF) technology. SPF is a forming method that produces complex shaped parts in a single forming process. The grain size, which is one of the most important superplastic parameters, affects the strain rate and ductility in grain boundary sliding (GBS). In GBS, a smaller grain size leads to a larger grain boundary area that results in higher elongation and therefore more favorable superplastic behavior.

In the present study, we concentrated on Zn-22Al, a commercial superplastic alloy having a wide range of applications in different fields of studies. Zn-22Al has a seismic property, which makes it applicable in buildings for energy absorption during earthquakes. Zn-22Al is also used for electronic enclosures, cabinets and panels, business machine parts, and medical and other laboratory tools. Zn-22Al has the characteristic of reaching its highest superplasticity at a relevantly lower temperature comparing other conventional superplastic alloys and can be used for superplastic forming and diffusion bonding purposes. However, applications of Zn-22Al receive some limitation especially in structural industries such as easily deformation under constant pressure due to its high strain rate sensitivity. Therefore, methods of improving mechanical properties of Zn-22Al, also developing new techniques for manufacturing new composites of this alloy would become essential.

The FSP technique, which is a relatively new processing method requiring fewer processing parameters, was used in the present study to refine the grain structure and improve the mechanical and superplastic properties of Zn-22Al. The results revealed FSP can achieve fine grains and improve the superplastic properties of Zn-22Al.

In addition, FSP was used for a novel forming method, known as friction stir forming (FSF), to produce multi-functional superplastic composites. By using the FSF technique, perforated steel sheet was mechanically interlocked between Zn-22Al sheets to produce a superplastic damping sheet with outstanding damping capacity. Moreover, insulated copper wire was mechanically interlocked within Zn-22Al alloy to produce a

multi-functional superplastic alloy with the ability to transfer information or energy. Finally, FSF can improve the mechanical properties of Zn-22Al superplastic alloy by interlocking the stainless steel strands within the alloy. Using the friction stir based technique can have a significant scientific impact on the production of functional composites with respect to both mechanical engineering and electrical engineering.

Contents

Overview of chapters

This dissertation consists of seven chapters. Each chapter is described as follows.

1. This study is mainly motivated by friction stir welding (FSW), a recently invented joining process that uses friction stirring to deform and join materials. An introduction is presented in Chapter 1. Friction stir welding, friction stir processing (FSP) and friction stir forming (FSF) as the bases of our study are explained in this chapter.

2. Chapter 2 contains a review of relevant literature. Reports from other scholars on this topic are described. The topics especially focus on the background of superplasticity, grain refinement techniques and applications of friction stir processing. Accordingly, Zn-22Al, the material used in our experiments, is introduced. Potential gaps in knowledge are identified and the need for current research is established.

3. Our study on grain refining of Zn-22Al superplastic alloy using FSP is presented in Chapter 3. It is discussed how FSP can affect and improve mechanical and superplastic properties of Zn-22Al alloy.

4. Chapter 4 describes a novel technique for producing a superplastic vibration damping steel sheet composite by using FSP.

5. Chapter 5 demonstrates a novel technique for mechanically interlocking Zn-22Al superplastic alloy to insulated copper wire to produce a multi-functional superplastic composite.

6. Chapter 6 proposes friction stir forming (FSF) for mechanically interlocking stainless steel strands to strengthen Zn-22Al alloy and to produce a composite with flexibility.

7. Finally, Chapter 7 outlines the main conclusions, and identifies both limitations to our study and recommendations for further research.

Bibliographic references cited in the present study are appeared in the reference list.

For better understanding of the dissertation structure, an illustration is presented in Fig. i below.

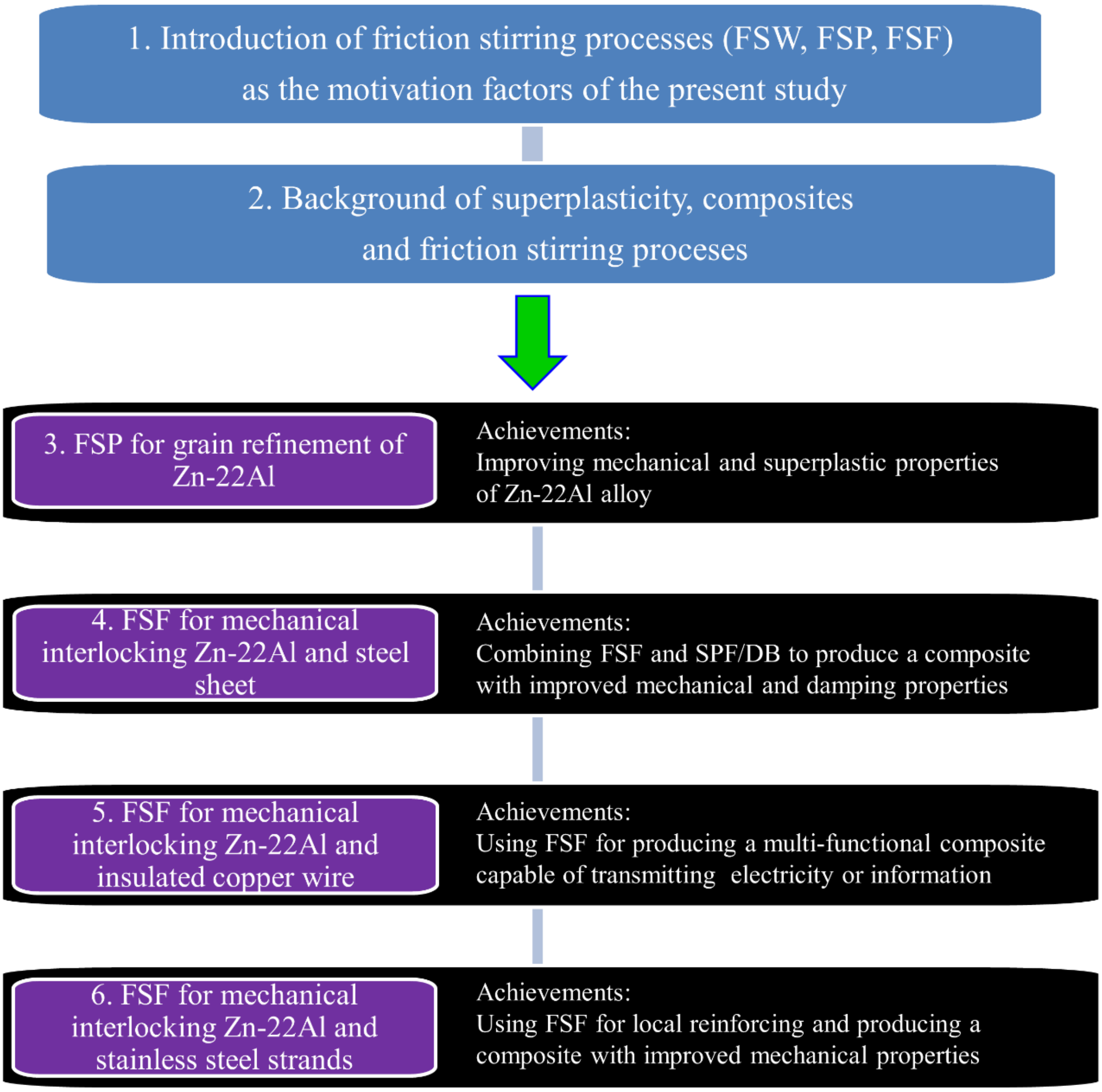

Fig. i Structure of the dissertation chapters

Chapter 1

INTRODUCTION

1. INTRODUCTION

1.1 Friction stir welding (FSW)

Friction stir welding (FSW) is a solid-state joining process that uses friction heat. This method was developed by TWI in Cambridge, England and patented by Thomas. [1] FSW overcomes many of the problems associated with traditional joining techniques. In particular, FSW produces high-quality welds in difficult-to-weld materials such as aluminum, so it is rapidly becoming the process of choice for manufacturing lightweight transport structures such as boats, trains and airplanes. In addition, FSW eliminates many of the defects, such as shrinkage, solidification cracking and porosity, in fusion welding techniques. Because friction stir welding is particularly appropriate for aluminum alloys, which often cannot be easily joined by standard welding methods, initial research efforts concentrated on these alloys.

Another important advantage of friction stir welding is that only a few process parameters need to be controlled. In fusion welding, many process factors need to be controlled: purge gas, voltage and amperage, wire feed, travel speed, shielding gas and arc gap. However, in FSW, only three process parameters need to be controlled: rotation speed, travel speed, and pressure, all of which can be easily controlled. Increased bond strength combined with reduced process variation increases the safety margin and the reliability.

Fig. 1.1 illustrates the principles of FSW. A cylindrical-shouldered tool with a probe is rotated, plunged and fed into the joint line between two pieces of sheet butted together. As the rotating tool is traversed through the joint, it generates frictional heat between the tool and the workpiece. The generated heat is sufficient to plasticize and form a solid-state weld in the base metal.

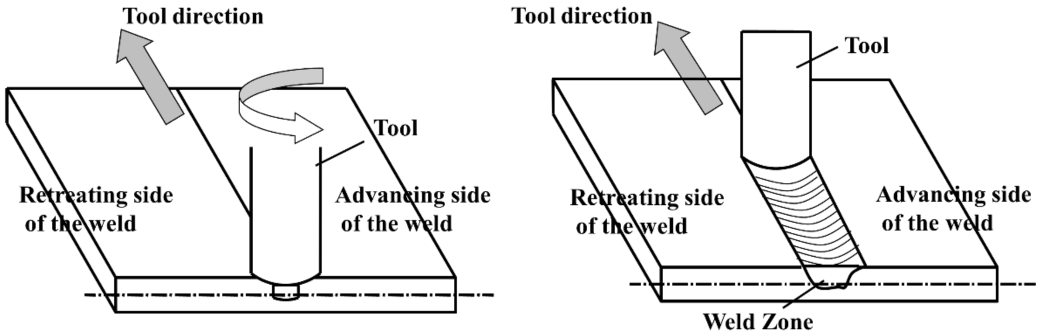

Fig. 1.1 Principle of friction stir welding

Fig. 1.2 shows the microstructure after FSW. Macro and microstructural investigations reveal that the friction stir weldment is composed of four different regions: 1. unaffected material, 2. heat-affected zone (HAZ), 3. thermo-mechanically affected zone (TMAZ) and 4. weld nugget or stirred zone. Unaffected material or parent metal is undeformed material located remotely from the weld. The unaffected material may experience a thermal cycle from the weld, but microstructure or mechanical properties are not affected by the heat. The heat-affected zone (HAZ) experiences a thermal cycle that modifies the microstructure and mechanical properties; however, no plastic deformation occurs in this area. The thermo-mechanically affected zone (TMAZ) is plastically deformed by the friction stir welding tool. The stir zone is the recrystallized area affected by stirring by the probe of the FSW tool.

The probe of the FSW tool is one of the most important parts of FSW equipment. The probe mainly performs the stirring function. The tool tilt angle and the plunge depth are other important parameters for FSW. Plunging the shoulder below the plate surface increases the pressure below the tool and helps ensure adequate forging of the material at the rear of the tool. Tilting the tool by 2–4 degrees has been found to assist this forging process. [2]

Fig. 1.3 shows a schematic illustration of the FSW tool used in our study. When the probe has a normal thread cut, the direction of rotation of the tool must be counter-clockwise so that the material does not jump onto the upper surface of the workpiece. In many cases, the shoulder part also has an angle. The angle, which is inclined in the concave direction from the outer periphery of the

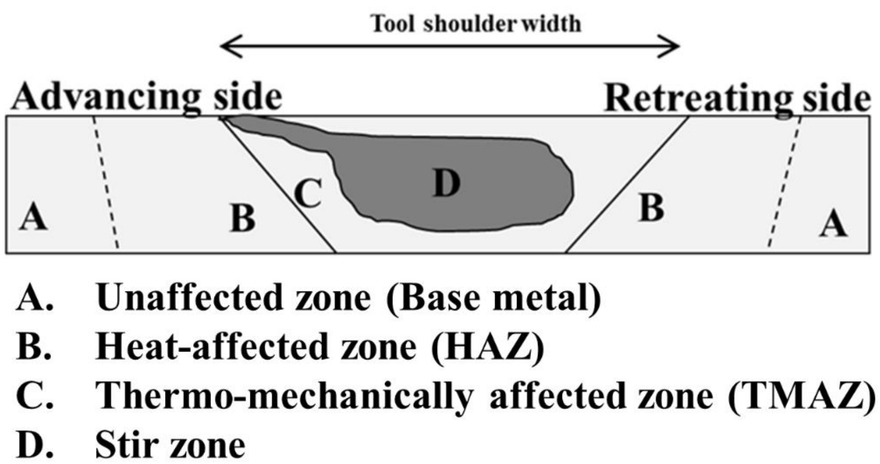

Fig. 1.2 Microstructure features after FSP

shoulder part toward the base of the probe, is expressed as negative or positive. When the inclination angle of the tool is set to 0 degrees and the shoulder angle is positive, an excellent bead can be obtained. However, when the tool inclination angle is inclined several degrees in the traveling direction, a negative shoulder angle can obtain a better bead appearance. Some research has reported examples in which a shoulder is provided with a spiral groove [3], but, generally, this research does not reveal the detailed shape.

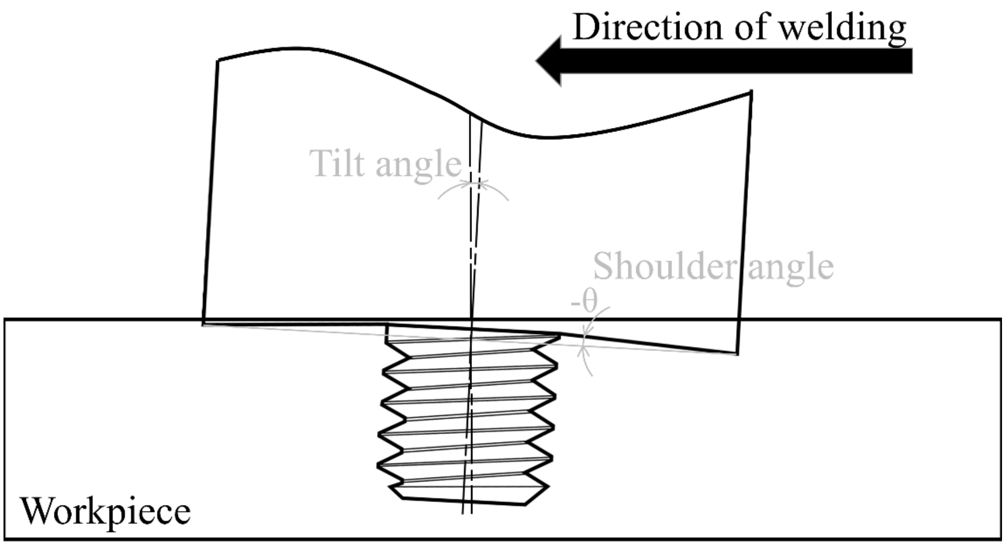

Fig. 1.3 Schematic illustration of friction stir welding tool used in our studies

1.2 Friction stir processing (FSP)

FSP uses the same methodology as FSW, but FSP is used to modify microstructures and does not join metals. The rotating tool provides continual hot working action that plasticizes metal within a narrow zone while transporting metal from the leading face of the probe to its trailing edge. The processed zone cools without solidification and forms a defect-free recrystallized, fine-grained microstructure. Essentially, FSP is explored as a thermo-mechanical metal working process that changes the local properties without influencing the properties in the remainder of the structure. By modifying the technology developed for FSW, FSP can be used to transform a heterogeneous microstructure into a more homogenous microstructure. FSP as a surface-engineering technology can eliminate casting defects and refine microstructures, and thereby improve strength and ductility, increase resistance to fatigue and corrosion, enhance formability, and improve other properties of the material. FSP also has the ability to thermo-mechanically process selective locations on the structure's surface.

FSP has recently gathered interest in many different fields of material studies, including superplasticity. A study has suggested that superplasticity occurs during the friction stirring process. [4] To impart superplasticity, FSP produces fine-grained microstructures. In one study, Nishihara et al. reported initial results on the FSW of superplastic Zn-22Al eutectoid alloy and demonstrated that FSW produces a fine-grained structure within the joint part. [5]

Zn-22Al is a well-known superplastic material that has been widely used in different fields of studies. Zn-22Al can be obtained as sheet for thermal forming and is often used in low-volume applications where tooling costs must be kept low. It is used for electronic enclosures, cabinets and panels, business machine parts, and medical and other laboratory instruments and tools. [6] However, few trials of applying FSP on this alloy have been conducted. In our laboratory, studies on controlling and improving the mechanical properties of this alloy have been conducted. In tensile testing under proper temperature conditions, the observed ductility of superplastic metal varies substantially with the strain rate. Significant losses in ductility occur as the strain rate is increased or decreased from the value at which the ductility is maximized. It is well known that the primary factor related to this behavior is the rate of change of the flow stress with respect to the strain rate, which is usually measured. The rate is referred to as m. Higher values of m correspond to greater superplasticity. [7]

One of the basic requirements for superplasticity is the material microstructure must have very fine grains. [7] It is expected that the grain refinement of Zn-22Al can be achieved through FSP. Such grain refinement would have great significance in superplasticity studies.

We focus on the effect of FSP on the microstructure of Zn-22Al superplastic alloy in Chapter 3. In particular, the effect of varying the different tool process parameters of FSP on the grain size of superplastic Zn-22Al is discussed based on microstructural observations, hardness tests and superplastic properties. The results of the study also open a new channel for discussing possible new applications and the challenges of FSW and FSP, such as applying FSP for improvement and development of fine-grained structures for increased superplasticity.

1.3 Friction stir forming (FSF)

Due to the fluidity of aluminum alloys during FSW, a new micro-forging method was developed and applied to mechanical interlocking. This process is referred to as friction stir forming (FSF). Nishihara et al. were the first to report FSF. They applied it to micro-forming and producing mechanical joints of aluminum alloy and steel. [8-10] The FSF process uses the friction heat and the plastic deformation generated between the rotating tool and the material being forged. Fig. 1.4, which was first published by Nishihara in 2003, explains the principle of FSF. A material plate is put on a die, and friction stirring is conducted on top of the material. The material deforms and precisely fills the narrow grooves of the die due to high pressure and plastic deformation caused by the friction stirring heat, and the shape of the die is reflected on the workpiece. FSF can be used for embossing or mechanically interlocking dissimilar alloys or even micro-form processing. This technique is now being studied in our laboratory.

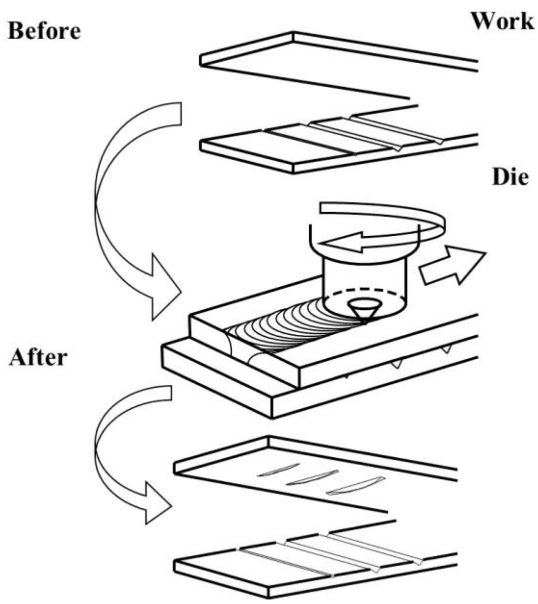

Fig. 1.4 Principle of FSF [9]

Yamamura et al. used FSF to mechanically interlock thin metallic wires to aluminum alloys. [11] Yamamura published an article about the excellent fluidity of aluminum alloys during FSP, and fabricated metal matrix composites (MMCs) by the local dispersion of aluminum oxide particulates in the aluminum alloy. [12] Yamamura et al. also proposed novel FSF methods, such as the fabrication of local metallic foam, for property modification of the MMCs of 6061 aluminum alloy. They dispersed titanium hydride particles (TiH_2) locally as a foaming material to produce the local MMCs. As a result, the structure of the foam produced in the layers was controlled by the titanium hydride particles dispersed in those layers. By monitoring the heat generated during FSP, they investigated the possibilities of dispersing the foaming material by the heat generated in FSP without additional heat from an electric furnace. The result showed the possibility of developing local foaming by the heat generated in FSF. [13]

FSF recently has been applied to material forming. Ohashi et al. used FSF for net-shape forming of A5083 aluminum alloy gear racks. [14] They used a commercially available gear rack as a die and put A5083 plate on top of the die. They applied FSF on the surface of the plate. As the result, the material was deformed and filled the die cavity.

In the present study, we used FSF for mechanical interlocking of dissimilar alloys. We experimentally investigated the mechanical interlocking of Zn-22Al superplastic alloy to a perforated steel plate and developed a superplastic vibration damping composite. Trials of interlocking Zn-22Al superplastic alloy to a thin insulated copper wire, as well as to stainless steel strands, to develop a new composite alloy are discussed in Chapters 4 to 6.

Chapter 2

BACKGROUND AND LITERATURE REVIEW

2. BACKGROUND AND LITERATURE REVIEW

2.1. Superplasticity and grain refinement

Superplasticity refers to the ability of a polycrystalline material to be pulled out in high tensile elongation without the development of necking prior to failure. [15] The original work on superplasticity was published by Pearson in 1934. [16] Pearson dramatically demonstrated a Bi-Sn sample that had been deformed to nearly 2000%. The ability to achieve high tensile ductility in a polycrystalline material is of interest from the point of views of scientific advances and the potential applications in the materials forming industry. [17] Superplasticity has several different variations of microstructural mechanisms and deformation conditions. These include micro-grain superplasticity, transformation superplasticity and internal stress superplasticity. At this time, only micro-grain superplasticity is important to the fabrication of parts. "For micro-grain superplasticity, high ductility is observed only under certain conditions, and the basic requirements are as follows: very fine grained material (on the order of 10 µm or finer), a controlled strain rate (usually 0.0001 to 0.1 s^{-1}), and a relatively high temperature (greater than approximately one-half the absolute melting point)." [18]

Many advanced materials including metal matrix composites, intermetallics and ceramics exhibit superplastic behavior. [19] Moreover, some very fine-grained materials exhibit superplastic behavior at a very high strain rate (higher than 1 s^{-1}). High strain rate superplasticity is very attractive for commercial applications, because one of the current drawbacks in superplastic forming technology is a slow forming rate, which is typically ~10^{-4} s^{-1}. Superplastic forming offers advantages over other fabrication methods. One of the major advantages of this process is that it can form large and complex workpieces in only one operation. The finished product has excellent precision and a fine surface finish. It also does not suffer from springback or residual stresses. Products can also be made larger to eliminate assemblies or reduce weight, which is critical in aerospace applications. [20] Superplasticity has been utilized for the fabrication of complex parts from sheet metal because of its large elongation. [21] A current goal in recent studies is the development of finer-grained materials that exhibit superplasticity at higher strain rates and lower forming temperatures.

Experimental evidence has shown that reducing the grain size increases the superplastic strain rate and superplastic elongation, and decreases the superplastic temperature. [22] The grain size affects grain boundary sliding (GSB), which is considered the main superplastic formation mechanism. [23]

2.1.1 Grain refinement methods

Recently, severe plastic deformation (SePD) techniques such as equal-channel angular pressing (ECAP) or torsion straining (TS) have been used to produce bulk materials with ultrafine grains. [24, 25]

The process of ECAP was first introduced by Segal et al. [26] The basic objective at that time was to develop a metal forming process where high strains are introduced into metal billets by simple shear. [27] After that, in the 1990s, Valiev et al. used ECAP to produce ultra-fine-grained metals with unique properties, and the resulting metals had a scientific impact on industrial applications. [28, 29] An important limitation in conventional ECAP is that the sample must be removed from the die and reinserted, with or without an intermediate rotation, in order to achieve large numbers of passes and a high imposed strain. These operations are both labor-intensive and time-consuming. A procedure that eliminates the need for removing specimens from the die between each pass is to using a rotary ECAP die. [30-33] However, a disadvantage of this process is that the aspect ratios of the sample are small, so the final effects can lead to significant inhomogenities. [34] A physically similar approach is the side-extrusion process. [35] A disadvantage of this method is that it requires the acquisition of a complex pressing facility. Nakashima et al. constructed a die for multiple passes as an alternative procedure. [36]

The principle behind these techniques is to produce a very large strain in the sample. The rearrangement of the dislocations introduced by straining leads to a substantial grain refinement down to the submicrometer or even the nanometer scale. Materials processed by ECAP have the potential for superplasticity, especially at very high strain rates and low temperatures. The validity of this proposal has been demonstrated by several recent reports of high strain rate superplasticity above 10^{-2} s^{-1} and low temperature superplasticity in aluminum and magnesium alloys processed by ECAP. [37-40]

Only a few reports are available on applying ECAP to sheet metals to produce ultra-fine grained material for industrial applications, where it is necessary for the as-pressed samples to be in the form of metallic sheets. [41-45]

However, another recent processing technique that has gathered interest for developing the mechanical properties of materials, especially sheet metals, is friction stir processing (FSP), which was introduced earlier.

FSP is believed to affect the grain size of the stirred material. Attempts to modify the structure of materials with FSP methods were carried out on Al alloys [46-48], magnesium alloys [49-54], steels [55], and also composites. [56]

Yong et al. reported the production of an ultra-fine-grained aluminum alloy with an average grain size of 2~3 μm by using FSP. [57] They applied single-pass FSP at various tool rotational speeds (560~1840 rpm) on 1050 aluminum alloy and investigated the influence of tool rotational speed on the microstructure and the mechanical properties of the zone. They set the tool travel speed to 155 mm/min. Their results showed a very fine-grained structure through the stirred area for a rotational speed of 560 rpm. The hardness was significantly increased by about 37% compared to the base metal. The tensile strength was also improved after FSP due to the grain refinement. They concluded that the maximum temperature of the processed area is an important parameter for controlling the microstructure and the mechanical properties. [58]

Hayashi et al. (2010) applied FSP on continuously cast AA5083 in the as-received condition. They reported grain refinement (1.0-3.5 μm) after FSP within the stir zone. They reported high elongation of 1200% at a high strain rate (10^{-1} s^{-1}) at a temperature of 450 °C. [59]

Haider et al. (2015) investigated the effect of FSP on the microstructure and the hardness of a 7000 Al alloy (Al-Zn-Mg-Cu) that was produced via casting with the addition of nickel. They performed FSP in a single pass with a rotation speed of 1500 rpm and a travel speed of 40 mm/min. A grain size of 18 μm with higher hardness was achieved in the stir zone. This grain refinement led to the uniform space distribution of Ni dispersed particles in the stir zone. [60]

Ehab et al. (2010) reported a study on trials of FSP on A5083 aluminum alloy. They applied FSP in a single pass with a rotational speed of 430 rpm and a travel speed of 90 mm/min. Their FSP treatment achieved a fine grain size of 1.6 μm and a strain rate sensitivity exponent (*m*-value) of 0.33 compared to the *m*-value of 0.018 before treatment. Tensile tests at a temperature of 250 °C revealed enhanced ductility and lower forming loads. [61]

Chengqi et al. reported that FSP was an effective method to enhance the Mg-Zr alloying efficiency and save the Zr addition. They used FSP to modify the Zr particle size distribution of a commercially available Mg-Zr master alloy and studied the subsequent grain refinement ability by trials on Mg-3Nd-0.2Xn-0.6Zr alloy. [62] Their study provided a more efficient way to achieve more Mg-Zr refinement. Their new method applied FSP to as-received Mg-30%Zr alloy in order to break up large Zr particles and clusters into smaller particles; they concluded that the refining efficiency of Mg-Zr alloy was improved by FSP.

Iwaszko et al. subjected AM60 magnesium alloy to FSP to improve its mechanical properties. They showed that FSP leads to a more homogeneous microstructure and significant grain refinement. They achieved an average grain size of 6-9 µm within the stir zone. They concluded that lower rotational speed of the tool resulted in a finer grain size and a higher hardness. They presumed that the temperature increases with the higher rotational speed led to a lower refinement of material. [54]

Charit and Mishra used FSP as a grain refinement technique. They applied FSP on a commercial 5083 Al alloy. Fine-grained microstructures with an average grain size of 3.5-8.5 µm were obtained in their study. They reported a maximum ductility of 590% at a strain rate of 3×10^{-3} s^{-1} at 490 °C. They indicated the importance of the processing window for obtaining an optimized microstructure for optimum superplasticity. [63]

Saito et al. applied a single pass of FSP (rotational speed: 1540 rpm travel speed: 0.5 mm/s) on rolled and annealed 1050 Al alloy and reported that grain refining by using FSP depends on the microstructure of the base metal. They achieved a grain size of 1-2 µm after FSP for the rolled plate, while the grain size of 4-8 µm was obtained for annealed sheets. [64]

McNelly applied multiple passes of FSP in a step-over method and reported significant grain refinement in AA5083 Al–Mg alloy. He used two different FSP tools with different probe lengths and conducted FSP with a step-over distance of 2 mm to obtain a grain size of 0.5-2.0 µm. He evaluated the processed alloy by tensile testing and measured a high elongation of 1200% at a high strain rate of 1×10^{-1} s^{-1}. [65]

Such discoveries in grain refining have enormous practical significance. For example, it was reported recently that ultra-fine-grained Zn–Al alloy could be applied as a seismic damper for high-rise buildings by attaining high strain rate superplasticity at room temperature. [66-70]

2.1.2 Grain refinement of Zn-22Al superplastic alloy

Zn-22Al, which is a well-known superplastic alloy used in our studies, can be obtained as sheet for thermal forming and is often useful in low-volume applications where tooling costs must be kept low. Zn-22Al is also used for electronic enclosures, cabinets and panels, business machine parts, and medical and other laboratory tools. [71] "Zn-22Al superplastic alloy also has excellent properties needed for seismic damper, such as high ductility, low work hardening and no metal harmful to human health." [71] Some investigations were carried out for nano-sized Zn–Al alloy in order to develop a high-performance seismic damper capable of replacing conventional dampers, such as low-yield-point steel. Using FEM analysis, Tanaka et al. discussed a design for appropriate structures as damping devices using superplastic Zn-Al alloy. They manufactured a bulk Zn–Al alloy with a nanocrystalline microstructure by thermo-mechanical controlling process (TMCP) technology, and compared the tensile properties of this alloy with that of low-yield-point steel. [72]

Only a few attempts have been made to improve the mechanical properties and to refine the microstructure of Zn–Al alloys by ECAP. [73-75]

Cetin et al. investigated the effect of grain size on the deformation behavior of Zn-22Al superplastic alloy. They produced a fine-grained structure having a grain size of 200 nm by applying the two-step ECAP method. They also applied an annealing process to the obtained alloy to achieve microstructures with various grain sizes ranging from submicron to micron sizes. The result showed 400% elongation at a strain rate of 5×10^{-2} s^{-1}. They also reported a lower strain rate with elongation decreased to 100%~390%, because the grain size increased due to annealing. [76]

In another study, Demirtas et al. reported the effect of natural aging at room temperature on the superplasticity of Zn-22Al alloy. They used two-step equal channel angular extrusion/pressing (ECAE/P) to produce ultra-fine grained Zn-22Al having a grain size of 200 nm. They subjected Zn-22Al to long-term aging at room temperature (up to 60 days). Grain sizes of 300 and 350 nm were obtained after 60 days of aging; this means that Zn-22Al has good microstructural stability at room temperature. The alloy subjected to ECAP showed a maximum elongation of 400% at a high strain rate of 5×10^{-2} s^{-1}. The elongation decreased to 350%-370% with the increase in grain size (300 and 350 nm). They concluded that examined Zn-22Al can be used in long-term applications at room temperature, as in the case of seismic conditions. [77]

Tanaka et al. reported a study on grain refinement of Zn-22Al in 2003. They subjected Zn-22Al to ECAE under different conditions. A grain size of 0.3-0.6 µm was achieved when 4 to 8 passes of ECAE were performed during alloy phase transformation at room temperature. They verified that equiaxed and homogeneously distributed grains were obtained by ECAE. [78]

Nishihara (2004) reported the initial results of the FSW of superplastic Zn-22Al eutectoid alloy. He demonstrated that FSW produces a fine-grained structure within the joint part. [79]

However, these studies showed disadvantages. For example, they are time-consuming and labor-intensive. The sample must be removed from the die and reinserted to achieve more passes in ECAE in order to obtain finer grains. In the present study, grain refinement of Zn-22Al is expected to be achieved by using FSP, which is time-saving, ecological, and financially cheaper, with easier experimental procedures; these characteristics could be of great significance in superplasticity studies. The effect of FSP on the microstructure of superplastic Zn-22Al is discussed in Chapter 3. In particular, the effect of varying the different tool process parameters of FSP on the grain size of superplastic Zn-22Al alloy is discussed based on microstructural observations, hardness test results and superplastic properties. The results of the present study also open a new channel for discussing possible applications and challenges of FSW and FSP, such as applying FSP to the improvement and development of fine-grained structures for increased superplasticity.

2.2 Composite materials

Composite materials have got a widely applications in cutting-edge ranges of advanced materials as medical devices, sport parts, automotive and especially for materials that are needed for aerospace, underwater, and transportation applications. Many of modern technologies require composite's properties that cannot be met by the conventional metal alloys, ceramics or polymeric materials. [80, 81] For example, aircraft engineers are increasingly searching for structural materials that have low densities, are strong, stiff, and abrasion and impact resistant, and are not easily corroded. This would rather be a combination of characteristics. [82] Material property combinations and ranges have been, and are yet being, extended by the development of composite materials. Generally speaking, a composite is considered to be any multiphase material that exhibits a significant proportion of the properties of each constituent phases such that a better combination of properties is achieved.

In designing composite materials, scientists and engineers have ingeniously combined various metals, ceramics, and polymers to produce a new generation of materials. Most composites have been created to improve mechanical properties such as strength, stiffness, toughness, and high-temperature strength. [81]

Many composite materials are composed of just two phases; one is termed as matrix, which is continuous and surrounds the other phase, often called the dispersed phase. The role of matrix in composite materials are to give shape to the composite, protect the reinforcements, and create toughness for material. The aim of reinforcements in composites are to get strength, stiffness and improve other mechanical properties.

The properties of composites are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase. [81]

Composites can be classified into different categories based on the type of matrix material. For example: metal matrix composites (MMCs) or fiber reinforced metals (FRM), fiber reinforced plastics (FRP), fiber reinforced ceramics (FRC), fiber reinforced glasses (FRG), intermetallic compound matrix composites (IMC), and carbon fiber reinforced carbon (CFRC) composite. [81] Based on the characteristic and properties of each different composite, they can be used in different field of studies. However, production of composites to give the expected properties is not simple. Many methods have been developed for fabrication of composite to meet specific design or manufacturing challenges. Selection of the appropriate method for a particular part is important and will depend on materials, the part design and end-use or application.

2.2.1 Metal matrix composites (MMCs)

When in a composite the matrix is metal, it is termed as “metal matrix composite” [82]. Some of the advantages of these materials over other matrix composites include higher operating temperatures, non-flammability, and greater resistance to degradation by organic fluids. Aluminium is one of the main and dominant choices of matrix material for majority of the MMCs. Titanium alloys have also a wide range of applications in MMCs. Titanium alloys have enhanced strength to weight ratios as well as improved strength retentions at 400-500 °C than those of aluminium alloys. Titanium MMCs are applied in applications where performance is challenged regardless of cost-efficiency [83, 84] The super alloys, as well as alloys of aluminum, magnesium, titanium, and copper, are employed as matrix materials. The reinforcement may be in the form of particulates, both continuous and discontinuous fibers, and whiskers; concentrations normally range between 10 and 60 vol%. [85]

Automobile manufacturers have recently begun to use MMCs in their products. For example, some engine components have been introduced consisting of an aluminium alloy matrix that is reinforced with aluminum oxide and carbon fibers; this MMC is light in weight and resists wear and thermal distortion. Metal matrix composites are also employed in driveshafts (that have higher rotational speeds and reduced vibrational noise levels), extruded stabilizer bars, and forged suspension and transmission components. The aerospace industry also uses MMCs. Structural applications include advanced aluminum alloy metal matrix composites; boron fibers are used as the reinforcement for the space shuttle orbiter, and continuous graphite fibers for the Hubble telescope. [85]

2.2.2 MMCs manufacturing methods

As mentioned earlier, MMCs have many advantages over metals or alloys.

Depending on the type and intended application of the composite, selection of the proper fabrication method is required.

There are several methods of producing MMCs like stir casting, squeeze casting, spray deposition, powder metallurgy, diffusion bonding and vapor deposition. Some of those important techniques can be categorize into the two groups of liquid-state processes and solid-state processes. A summary of fabrication techniques are provided here.

2.2.2.1 Liquid-state processes

Casting or liquid infiltration implies infiltration or particulate reinforcement preform by a liquid metal. Poor wetting of ceramic reinforcement by the molten metal during liquid-phase infiltration results many challenges during MMC fabrication. Reactions between the fiber and the molten metal, which break down the properties of the fiber, are likely to happen when the infiltration of a fiber perform takes place. Applying fiber coatings before infiltration improves wetting and restrains interfacial reactions. However, the drawback is that the fiber coatings must not be exposed to air prior to infiltration because of the risk of surface oxidation of the coating. [86, 87]

Another liquid infiltration process in which ceramic particles and ingot-grade aluminum are mixed and melted to form a MMC is *Duralcan process*. [87] Melted mixture is stirred slightly above the melting temperature (600−700 °C). This technique requires particles with size range between 8−12 μm. In foundry-grade MMCs, high Si aluminum alloys (eg, A356) are used, in order to avoid the formation of the brittle compound aluminum carbide Al_4C_3, which is formed from the interfacial reaction between Al and SiC. Al_4C_3 is extremely detrimental and tend to harm the mechanical properties, especially toughness and corrosion resistance. Fiber tows are passed through a molten metal bath, where individual fibers are wetted by the molten matrix removed off from the excess metal, and a continuous fiber reinforced MMC is produced. [87]

Lanxide's primex process is another liquid metal infiltration process for producing MMCs, which can be utilized with particular reactive metal alloys such as Al-Mg that infiltrate ceramic preforms. For an Al- Mg alloy, in a nitrogen-intense environment, the process occurs between 750−1000°C, and standard infiltration rates are less than 25 cm/h. [87]

Squeeze casting or pressure infiltration refers to driving a liquid metal into a fibrous or particulate preform. [88] As result of the pressure upon completion of solidification, molten metal passes through small aperture in the fibrous preform, so that excellent wettability of the reinforcement by the molten metal will not be required. The process time in this technique is substantially short. Therefore, the reaction between the reinforcement and molten metal in the produced composite is minimized. Conventional casting defects such as porosity and shrinkage cavities are barely observed in these types of composites [83, 89].

2.2.2.2 Solid-state processes

"*Deformation processing* is a solid state technique in which the composite material is deformed and/or densified. Mechanical processing (swaging, extrusion, drawing, or rolling) of a ductile two-phases metal−metal composite triggers the two phases to co-deform, leading to one of the phases to elongate and become fibrous in nature within the other phase. The materials produced are occasionally denoted as in-situ composites. The characteristics of the preliminary materials define the properties of a deformation processed composite. The initial materials are normally a billet of a two-phase alloy that has been made by casting or powder metallurgy methods" [83, 90].

Roll bonding is another conventional practice to produce a laminated composite. The produced composite consists of distinct metals in layered arrangement which is called sheet laminated metal-matrix composites [91].

"*Powder processing* techniques are utilized to make particulate or short fiber reinforced composites in association with deformation processing. Generally, this method includes cold pressing and sintering or hot pressing to manufacture primarily particle or whisker-reinforced MMCs." [92] The matrix and the reinforcement powders are mixed together to create a uniform distribution. Cold pressing is applied accordingly to construct a so called green body which is about 80% dense and can be simply processed. "To eliminate any absorbed dampness from the particle surfaces the cold pressed green body is preserved in a sealed container and degassed." [83] The material is hot pressed either uniaxially or isostatically and extruded in order to achieve a completely dense composite. The stiff particles or fibers trigger the matrix to be deformed considerably. Furthermore, dynamic recrystallization at the particle/matrix interface during hot extrusion generates randomly oriented grains near the interface, and moderately textured grains far from the interface. [92]

Sinter-forging is a unique and economical deformation processing technique [93]. “In this method a powder mixture of reinforcement and matrix is cold compacted, sintered, and forged to practically complete solid. The major benefit of this technique is that forging is carried out to deliver a near-net shape material, leading to minimize the machining operations and material waste. Tensile and fatigue properties of the low cost, sinter-forged composites are equivalent to those of materials produced by extrusion.” [83]

Diffusion bonding is a solid-state processing method for merging similar or dissimilar metals. The bonds form as a result of inter-diffusion of atoms between in-contact metallic surfaces at high temperature. “As the main advantages, this technique is able to process a broad range of metal matrices and to control fiber orientation and volume fraction.” [83] However, long processing times, high processing temperatures and pressures that leads to a costly process, and a restriction on producing complex profiles are the demerits and drawbacks of this method. Almost all forms of diffusion bonding processes simultaneously comprise application of pressure and elevated temperature. In this method, “matrix alloy foil and fiber arrays (composite wire) or monolayer laminate are pressed in a prearranged order. Vacuum hot pressing is critical phase in the diffusion bonding processes for metal matrix composites. As an alternative of uni-axial pressing, hot iso-static pressing (HIP), may also be applied in which the composite inside the container is consolidated via the gas pressure against a can. The HIP facilitate applying high pressures at high temperatures with inconsistent geometries.” [83, 94]

With knowledge of various types of composites, as well as an understanding of the dependence of their behaviors on the characteristics and properties of the constituent phases, it would be possible to design materials with property combinations that are better than those found in the metal alloys, ceramics, and polymeric materials. [82]

2.2.3 Superplastic metal matrix composites

As indicated, composites are strengthened by particles or fibers which in many cases make them have a low ductility. It produces problems in forming process and limits their widespread applications such as preventing mass production through press or rolling methods, resulting in high costs. Therefore, improvement ductility of composites and producing fine-grained MMCs has been desirable for many structural applications. For commercial application, development of manufacturing technology and forming operations of the composites possessing high ductility is required. [95] Therefore, there has been a growing interest in the development of fine-grained microstructures for superplastic forming of different alloys matrix composites.

Some studies have been performed to develop superplastic ceramic reinforced aluminum composites with high ductility. Superplastic composites have been processed by foils, casting, hot rolling or the powder metallurgy method. Kojima and Ishikawa used hot rolling technique to produce a superplastic aluminum sheet clad with metal matrix composites. [96] They fabricated a clad sheet by the hot rolling of an aluminum alloy sheet and a TiN-particle-reinforced aluminum composite sheet which were fabricated by powder metallurgy and thermos-mechanical processing. Fabricated clad sheet indicated an elongation of 257% at a strain rate of 8.3×10^{-3}. [96] Their method have the advantage of improving the superplastic properties of the assembly by refining the microstructure of the composite throw the thermo-mechanical process following the powder metallurgy. However, the processing method was complicated with many parameters to control.

Nishimura et al. published a study on manufacturing the Zn-22Al superplastic matrix composite by using powder metallurgy. [97] They used bondable Zn-22Al alloy powders and SiC fibers to develop a compacting method for fabricating metal matrix composites. They applied heat treatment to control the composite properties. They manufactured metal matrix composites by the vacuum hot press method. The maximum applied pressure was 50 kN and the maximum heating temperature was 600 °C. A cylindrical mold (⌀ 70, 70 mm × 50 mm) was used in their experiments. Because the reinforcing fiber was supplied with SiC continuous fiber in 500 bundles, it could be cut to an appropriate length according to the size of the mold. Cut fibers and superplastic powder were placed alternately and set under the appropriate conditions with a hot press. The pressure was set to avoid damaging the fiber, and the retention time was set to the minimum value obtained from various experiments. The

FRM molded under the above conditions was then divided into those subjected to heat treatment (360 °C× 1 h → rapid cooling) to induce superplasticity and those not subjected to heat treatment (referred to as non-heat treatment).

The method of Nishimura et al. required control of the fabricating temperature, the amount of applied pressure, and also the duration time. Consequently, their method is not efficient enough because it is too time-consuming, complicated and not eco-friendly.

Few studies have examined grain refining and development of superplastic characteristics in aluminum composites, because superplastic forming of aluminum alloys especially have been demonstrated in some aluminum based composites. [98-100] Mabuchi and Imai published a study on producing a superplastic composite reinforced with Si_3N_4 by powder metallurgy. [101] They used the powder metallurgy method and produced a homogeneous dispersion of the reinforcement (Si_3N_4) and the fine-grained structure in an aluminum matrix composite. They reported large elongation of 300% at a high strain rate (4×10^{-2}). It was reported that 2124 aluminum composite containing 20% SiC whisker exhibited about 300% elongation to failure [102] Mahoney and Gosh fabricated superplastic 7064 aluminum alloy reinforced with 10% SiC particles. [103-105] A SiC reinforced 2124 aluminum composite which was fabricated by the powder metallurgy method, showed a large elongation of about 300% at a high strain rate. [102] Hot extrusion method has also been noted as a method for processing superplastic aluminum alloy composite without any thermomechanical treatment for grain refinement. [106]

However, each of the superplastic composites process methods like foiling, casting or powder metallurgy, have some problems and disadvantages. Fabrication process of composite with superplasticity is complex. Additional thermo- mechanical processing (TMP) is needed for mixing processes. TMP is a complicated method containing warm rolling, which leads to high cost. [95, 107]. Range of deformation is slow and it is not relatively suitable to apply for superplasticity. Moreover, “range of deforming temperature is above the solidus line of matrix; liquid-and-solid coexistence causes voids, which degrade mechanical properties of the products.” [95]

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2.2.4 SPF/DB for manufacturing metal matrix composites

Superplastic forming /diffusion bonding (SPF/DB) is relatively a new method for fabricating composite sheets and selectively reinforced composites. [108, 109] SPF and SPF concurrent with diffusion bonding (SPF/DB) of metals have been used in different industries such as the electronic, transportation, architecture, medical and mainly and especially in aerospace industry.

The main characteristics of the diffusion bonding process are very similar to those of SPF [110]. The similarity of the technological parameters for both technologies makes it possible to combine SPF and DB and thus highly complex structures are superplastically formed and diffusion bonded within the same heat-cycle [110]. Superplastic forming and diffusion bonding processes operate similarly to hot forming processes with a few distinctions. They still rely on the fundamental principle of heating the metal to increase its malleability. Superplastic forming requires exceptional amount of heat for the metal to reach its superplastic state. Once certain materials reach close to their phase transformation temperature, the material is so malleable that superplastic forming processes can use gas pressure to form the part. This allows operators increased control to form more complex parts than possible with hot forming.

Diffusion bonding processes form two or more sheets of metal into one part. It is accomplished by applying gas pressure between the sheets and around shapes once the metal has reached the super plastic state. Diffusion bonding processes have a variety of applications since they can form complex shapes.

Materials for SPF/DB processes should have good superplastic properties including elongation, strain rate sensitivity and cold ductility. In addition to the superplastic properties, the material to be formed must be suitable for diffusion bonding. "Diffusion bonding refers to the solid-state joining of the surfaces of similar or dissimilar metals by applying heat and pressure for a time duration to cause co-mingling of the atoms at the joint interface." [111] These properties make it possible to fabricate components from superplastic materials.

Significant cost saving in SPF/DB results from the shorter time required in forming and lower costs of the processes and metals used. [112]

High strength materials such as titanium have the most manufacturing applications in many industries like offshore and aerospace; therefore, the structure formed by the SPF/DB process provides higher rigidity and strength-to-weight ratio than conventional riveting assemblies, due to a large reduction in the number of components and higher structural efficiency. [112]

Generally titanium (Ti-6Al-4V alloy) and aluminum alloys (7475 alloy) which are resistant to cavitation with very fine grain size usually less than 20 μm are used as superplastic matrices. Hefti Larry carried out superplastic forming (SPF) of ordinary Ti-6Al-4V alloy at 900 to 920 °C using powders having an average particle diameter of 8 μm, and developed a technique for SPF at 775 °C. They also clarified that diffusion bonding can be performed at this relevantly low temperature with the same time and pressure as for conventional alloys. This microcrystalline grain Ti-6Al-4V was also capable of bonding with the conventional Ti-6Al-4V. They showed application examples of aerospace products using fine grain Ti-6Al-4V through joint research with VSMPO and Boeing. [113]

Velicki introduced a cost efficient method for making a SPF/DB hollow core fan blade. [111] A hollow core fan blade for a gas turbine engine, having a continuous leading edge, was fabricated using a four-sheet superplastic forming/diffusion bonding process which resulted in a cost-efficient and lightweight, yet strong, structure. The rotor blade was comprised of a face sheet which had a 180 degree bend therein so that the two face sheet ends were aligned. To fabricate the blade, a core sheet assembly was inserted inside the prepared face sheet; thereby a titanium pack assembly having a plurality of pressure-tight cells was formed. The titanium pack was inserted into a cavity within a die, after which the rotor blade, having predetermined design characteristics, was superplastically formed by heating the die and selectively pressurizing the plurality of cells. [111]

Zhihao et al. fabricated the multilayer structure of Ti_2AlNb based alloy based on the superplastic forming/diffusion bonding (SPF/DB). [114] They investigated properties of superplasticity and diffusion bonding of Ti-22Al-27Nb alloy. In their study the uni-axial tensile experiment showed that the tensile elongation reached up to 236% at the temperature of 970 °C with the strain rate of 3^{-4} s^{-1}. Based on the superplasticity, the finite element analysis of structural design was carried out and the results showed that the honeycomb structure was flexible. Diffusion bonding experiment was carried out at the temperature of 950 and 970 °C. Combining the microstructure of bonding interface with shear strength of different joints, the diffusion bonding parameter for present alloy was:

970 °C/ 10 MPa/ 2h. The thickness distribution of the final structure was uniform and the mechanical properties after SPF/DB reduced. The compressive strength of this structure behavior was about 7.7 MPa. [114]

Hirobashi et al. tried joining experiments by SPF/DB method with three kinds of superplastic aluminum alloys as the target and examined the possibility of diffusion bonding by carrying out shear test. In their study ultra-duralumin type A7475 and corrosion resistant aluminum alloy A5083 reached practical level even in low vacuum or argon gas. However, Zn-22Al did not have a high bonding strength. [115]

Nagano and Wakai publish a study on the possibility of using SPF/DB as a manufacturing technique in ceramics industries. [116] As recent developments of superplastic ceramics have brought various possibilities for manufacturing techniques in ceramic industry, they showed the possibility that the similar developments can be anticipated throw SPF/DB also in ceramic technology. Their study gave an overview on fundamentals and characteristics of SPF/DB method, and made a comparison between techniques for metals and that for ceramics. The diffusion bonding of ZrO_2/$Al2_2O2_3$ composites, mullite/ZrO_2 composite, and Al_2O_3/TiN composite was described as examples. The fabrication of functionally gradient material was demonstrated as an application of SPF/DB for ceramics. [116]

As indicated above, development of superplastic materials has provided new opportunities for designing and producing complex components and structures using superplastic forming methods. Superplastic forming methods can be used alone or in combination with diffusion bonding as an attractive technology for manufacturing of components and structures that cannot otherwise be produced.

As mentioned earlier, the materials that can presently be used in SPF/DB processes are limited to uniform grain-size metals that do not form an inhibiting surface oxide, thus, titanium alloys are the most favorite alloys for SPF/DB and it would be highly desirable to be able to form composite structures from aluminum and other aluminum alloys by SPF/DB technology.

Comparing to materials that exhibit uniform fine grains and superplasticity, Zn-22Al which is of the most commonly used materials for studying superplastic behavior, reaches its superplastic temperature at a relevantly low temperature between 200 and 300 °C and would be an excellent material for SPF/DB processes. However due to its low strength the structural applications of this alloy are limited. Only a few studies have been published regarding fabrication of Zn-Al alloys to produce applicable superplastic composites.

Azujima successfully developed a composite damping steel plate laminated with steel sheets. [117] He introduced a production method, characteristics and optimum design for low carbon steel sheet and Zn-22Al alloy.

Kaneko et al. reinforced Zn-22Al with SiC whisker throw high-pressure casting and hot extrusion methods, and observed the whisker breakage situation and investigated mechanical properties of the produced assembly. Although during the time of hot extrusion of the composite material the deformation resistance decreases, they could confirm and clarify that the whiskers can be uniformly distributed. Breakage by extrusion processing was reduced, and it could be oriented in one direction. They showed that elastic coefficient and the tensile strength of the extruded composite material were improved. [118]

Kim et al. produced a composite in which whiskers are three-dimensionally and randomly oriented in Zn-22Al alloy by combining high-pressure casting and hot extrusion methods. They applied eutectoid transformation process to form fine grain structures, and investigated the tensile properties and high temperature deformation behavior of the produced assembly under different strain rates in a temperature rate of 220~300 °C. They found out that the composite was influenced by the length and orientation of whiskers. However, even under conditions in which the matrix material exhibited superplasticity, the composite did not show superplasticity and broke at 100% or less elongation. [119]

Uno et al. prototyped a cylinder head gasket of composite material using a Zn-22Al based superplastic alloy and applied the composite as a material for sealing parts. [120] The prototype gasket for small diesel engines has a structure in which a Zn-22Al superplastic material is thermally crimped on both surfaces of a wire mesh or a fractured composite steel plate as a core material. They applied heat treatment after molding. As a result of various characteristic tests, the composite material using Zn-22Al based superplastic alloy was able to reduce creep deformation under high temperature and high pressure by using a composite material while maintaining its merits. As a result, they concluded that the developed composite can be used as a gasket material. [120]

2.3 Friction stir processing and its applications

2.3.1 Friction stir processing for fabrication of composites

As mentioned earlier, friction stir processing (FSP) is a new solid-state technique that uses the principles of friction stir welding. Friction stir processing results in a very fine and equinox grain structure in the processed regions. This grain structure causes higher mechanical strength and ductility. The microstructure in the processed region evolves through a continuous dynamic recrystallization process [121, 122]. FSP research has reported on refining grain, improving mechanical properties and developing superplasticity characteristics. [123] FSP has also been applied to formability improvement, surface treatment, and surface-modified powder processing. [124] Furthermore, material with grain refinement produced by this process exhibits superplastic properties. [125] Initially, FSP was used to modify the microstructure of cast aluminum alloy. Mahoney et al. [126] emphasized that FSP can be selectively applied to a location within a conventional aluminum alloy sample to tailor its microstructure and to achieve increased superplasticity. Mahoney and Lynch reported practical applications of FSP, including the application of FSP to Al-, Cu-, Fe-, and Ni-based alloys to improve their material properties. Some of the demonstrated beneficial effects of FSP include the following: doubling of the strength of cast Ni–Al bronze, a five-fold increase in the ductility of Al alloy A356, increased fatigue life of fusion welds, increased corrosion resistance of a Cu–Mn alloy, and bending of 25-mm-thick Al alloy 2519 plate to an angle of 85° at room temperature without surface cracking. [127]

FSP has been recently used to fabricate bulk and surface composites. The method of composite material formation using FSP was first reported by Mishra in 2003. Mishra used SiC powder as a reinforcing agent and 5083 aluminum alloy as a base material. The SiC powder was mixed with a small amount of methanol and attached to the surface of the aluminum alloy to form a SiC particle layer on the surface of the alloy. After that, FSP treatment was performed to disperse the SiC particles in the aluminum alloy and compositing was successful. [128]

Production of a surface layer composite by using FSP was reported by Lee et al., who made a groove in AA61 magnesium alloy and put SiO_2 nanoparticles in it. They restored the surface of the groove by performing FSP on the surface. FSP was performed again (multi-pass FSP) to disperse SiO_2 particles by AZ61 to form a composite material layer. They reported that the prototype composite material exhibited a high elongation of 420% and superplasticity when subjected to a tensile test at 350°C. [129]

Lim et al. reported that complex layers were formed on A7075 aluminum alloy by using multi-wall carbon nanotubes (MWCNTs). [130] They made grooves in A7075 and inserted MWCNTs into the grooves. 6111 aluminum alloy was overlaid on top and FSP was performed to form a composite material layer.

Dixit and colleagues also dispersed NiTi powder in A1100 in the same way to form a surface composite layer. [131]

However, when fabricating a composite material by using FSP, the reinforcing particles are not uniformly dispersed. It is thought that this is due to the non-uniform plastic flow of FSP. Therefore, Shafiei et al. performed FSP in several passes, called “multi-pass FSP”, in order to uniformly form the layer of a composite material. They dispersed Al_2O_3 into A6082 by using a tool without a probe and then repeated FSP up to 4 passes by using a tool with a probe. As a result, they found that by increasing the number of passes, the reinforcing particles were uniformly dispersed in stir zone (SZ) and the hardness was increased. At the same time, they reported that, by using nano-sized Al_2O_3 and the pinning effect, they could regulate the regrowth of the aluminum alloy structure refined by dynamic recrystallization during the FSP process and obtain crystal grains of 300 nm or less. [132]

Asadi et al. analyzed the effect of the number of passes of multi-pass FSP on the particle dispersion, which strengthens the surface layer of a composite. They also studied the effect of multi-pass FSP on the grain size of SZ. They dispersed Al_2O_3 and SiC powder in AZ91 magnesium alloy and applied FSP up to 8 passes and evaluated the results. Al_2O_3 and SiC were uniformly dispersed in 8 passes, and grains were reported to be refined to 1.3 μm for 50 μm Al_2O_3 and 800 nm for SiC. [133]

Many studies have reported on the use of FSP techniques for the fabrication and strengthening of composite materials by dispersing reinforcing particles. Using this technique, Hangai et al. produced porous aluminum precursors by dispersing TiH_2 in aluminum. [134]

2.3.2 Development of functional composites by using FSP

Manufacturing methods of various functional composites and foaming materials have been developed and studied, as described earlier. In current manufacturing studies, it is necessary to use a large heat source and an expensive matrix. Therefore, FSP is attracting great interest as an energy-saving technology in composite manufacturing, material modification, and formation of composite materials. Beside the benefits of FSP as a manufacturing method, it can also be considered as a novel forming technique. As indicated, FSP was introduced by Nishihara as a forming method for the first time as friction stir forming (FSF). [9] Considering the cost of reinforcement particles in the manufacture of composite materials by FSP, and the fact that dispersing particles within the whole material is not necessarily required, Yamamura et al. reported that FSP is an effective reforming process to impart functions to only necessary parts. Focusing on local functions, they strengthened necessary parts by actually forming a composite material locally. They proposed a method of locally forming a metal foam. They also proposed a local reforming method of aluminum alloy by using FSF. [135] FSP, which controls material characteristics, has also attracted attention as a manufacturing method for composite structures. The previous section focused on these studies and characterized the features.

Metal wires have been used in a wide range of fields due to their excellent strength and conductivity of heat or electricity. Metal wires are also used as a reinforcement of composite materials in various matrix composites. Fiber reinforced material (FRM) using thin metal fibers (wires) has been developed for metal reinforcement. FRM has gathered attention because of its beneficial properties, such as heat resistance, abrasion resistance and specific strength. Morooka conducted a powder rolling method to produce fiber reinforced composite material not only at a lower temperature in comparison with the molten metal infiltration method but also in a shorter time in comparison with the hot pressing method. [136] He used stainless steel wire and piano wire as the reinforcing fiber and pure aluminum powder as the matrix. Then, he simultaneously rolled powder and steel wire and succeeded in manufacturing steel wire reinforced aluminum. However, regardless of the excellent properties of FRM, a few problems and challenges remain. One problem that makes it difficult to produce FRMS is the brittle compound that sometimes occurs at the interface between the fiber and the metal matrix.

Many FSP studies have investigated the preparation of composite materials to increase strength, but studies are also underway to bring new functions to materials. Yamamura et al. produced a prototype of a new functional composite material by embedding copper wire, piano wire and optical fiber in an aluminum alloy by using FSF technology developed at Kokushikan University. [11] They cut a slit in aluminum alloy A6061 and put different wires inside the slit. They applied FSP on the surface of the plate. The softened material flowed and precisely filled the slit while wire with the aluminum alloy were mechanically interlocked.

However, only a few number of studies have fabricated superplastic alloys. Additionally, no studies have reported fabrication of functional Zn-22Al superplastic alloy using friction stirring process.

In the present study, we focus on fabrication of Zn-22Al superplastic alloy and produce a multi-functional superplastic alloy by using FSP and FSF techniques. The novel idea of producing a superplastic damping-vibration composite by FSP, which also has an effect on improving the mechanical properties of material, is discussed in Chapter 4.

Fabrication of Zn-22Al alloy by using FSF and production of a multi-functional superplastic alloy capable of transmitting data or energy is discussed in Chapter 5.

Finally, we discuss FSF as a novel method to mechanically interlock stainless steel strands with Zn-22Al alloy and produce a superplastic matrix composite. This is presented in Chapter 6.

2.4 Differences between conventional research and this research

Many studies have been published regarding grain refinement and fabrication of different alloys and composites. There are numerous methods for fabricating composite components. As discussed earlier, ECAP is one of the most popular methods of treating alloys to obtain fine grains and has been introduced as one production method for superplastic MMCs. However, the amount of time and high expense have been disadvantages. FSP as a new processing technique has recently gathered interest for fabricating alloys. Using friction stir processing

(FSP) for fabrication of alloys is quite recent. FSP as a direct solid-state processing technique attains microstructural modification. This technique has low cost and eco-friendly advantages since there is no harmful gas or radiation and noises because the heat generated during FSP comes from friction and plastic deformation. By optimizing the FSP tool design and parameters, microstructures and mechanical properties of the processed zone can be controlled. While it is hard to reach an optionally accustomed processed depth using conventional metal working procedures, the depth of the processed zone can be optionally managed by changing the length of the tool probe. FSP makes it possible to improve the properties of material partially and can create a partially functional material. Moreover, FSP is of great interest because it has the potential of refining the grain size of material and thus can promote superplastic flow at a high strain rate. This means a more complex part can be fabricated through superplastic forming processes.

Selection of a method and technique for a particular part, depends on the materials, the part design and final use or intended application, therefore, new methods may need to be developed to meet specific design or manufacturing challenges.

Zn-22Al alloy, a most commonly used material for superplasticity studies, with excellent damping properties, reaches its superplastic temperature at relevantly low temperature between 200 and 300 °C. These characteristic of Zn-22Al makes it an excellent material for superplastic forming processes combined with diffusion bonding. However, Zn-22 Al is highly strain rate sensitive and easily deforms when subjected to a constant temperature and constant stress. Because of that reason, it has only a few applications in the structural industry. Therefore, there is a need to defeat this weak points by improving the mechanical properties of Zn-22Al alloy or by producing and developing its composite alloys. However, as indicated above only a few studies have been published on the development of superplastic composite alloys based on Zn-22Al. In present study FSP is applied to Zn-22Al in a novel method and it is demonstrated that by controlling the process parameters of FSP tool, the superplastic phenomena temperature of Zn-22Al can be reached, therefore, by combining FSP and SPF/DB, new functional composites can be produced which would be difficult to produce by other conventional techniques. It is demonstrated that FSP can provide innovative functions for the superplastic alloy, also improve the mechanical properties of the manufactured superplastic composite while still keeping the superplastic properties of the alloy. This can suggest new applications of this fabricated superplastic composite and have an impact in opening new possibilities in engineering fields.

For more clarity and better understanding, reports of related studies are listed in Tables 2-1 to 2-2. Some disadvantages and differences are shown by comparing our studies and the other studies.

Table 2.1 Reports of superplasticity and grain refinement (No. 1)

Alloy or composition	Processing method	Grain size	Superplasticity			Reference	Difference or disadvantage of the study compared to our study
			Testing temperature (°C)	Strain rate (s^{-1})	Maximum elongation (%)		
Zn-22Al	ECAP, 8 passes	0.4-0.8 μm	150 200	3.3×10^{-3} 3.3×10^{-2}	940 1970	Furukawa et al. (1998) [137]	Labor-intensive and time-consuming
Zn-22Al	ECAP, 12 passes	0.6 μm	260	1.0	>2380	Lee & Langdon (2001) [138]	Labor-intensive and time-consuming
Zn-22Al	ECAP, 2 passes	200 nm		5×10^{-2}	400	M.E. Cetin et al. (2016) [76]	Labor-intensive and time-consuming
Zn-22Al	ECAE/P, 2 passes	200 nm	Room temperature	5×10^{-2}	400	M. Demirtas et al. (2016) [77]	Labor-intensive and time-consuming
Zn-22Al	ECAE	0.35-0.6 μm	Room temperature to 100			T. Tanaka et al. (2003) [78]	Labor-intensive and time-consuming
AM60	FSP, single pass	6-9 μm				J. Iwaszko et al. (2016) [54]	Non-threaded tool
A1050	FSP, single pass	2-3 μm	Room temperature		41	Y.J. Kwon et al. (1993) [57]	Non-threaded tool
A1050	FSP, single pass	3-4 μm	Room temperature		140	Y.J. Kwon et al. (2004) [58]	Non-threaded tool

Table 2.1 Reports of superplasticity and grain refinement (No. 2)

Alloy or composition	Processing method	Grain size	Superplasticity			Reference	Difference or disadvantage of the study compared to our study
			Testing temperature (°C)	Strain rate (s^{-1})	Maximum elongation (%)		
Mg-Zr	FSP, step-over multi passes	77-362 µm				C. Wang et al. (2014) [62]	Grain refiner is used, non-threaded tool probe
AA5083	FSP, multi-passes	1.0-3.5 µm	450	1×10^{-1}	1200	J.T. Hayashi et al. (2010) [59]	Overlapped multi-passes, non-threaded tool
A7075	FSP		730-510	1×10^{-1}	1000	Mishra and Mahoney (2001) [139]	
Al-Zn-Mg-Cu +5%nickel	FSP, single pass	18 µm				H. T. Naeem et al. (2015) [60]	No angle in tool shoulder
A5083	FSP	1.6 µm	250			E.A. Al-Danaf et al. (2010) [61]	
A7075	FSP + rapid cooling	100-500 nm				J. Q. Su et al. (2006) [140]	

Table 2.1 Reports of superplasticity and grain refinement (No. 3)

Alloy or composition	Processing method	Grain size	Superplasticity			Reference	Difference or disadvantage of the study compared to our study
			Testing temperature (°C)	Strain rate (s^{-1})	Maximum elongation (%)		
A5083	FSP	6.5 µm	530	3×10^{-3}	590	Charit and Mishra (2004) [63]	
A1050	FSP, single pass	1-2 µm				N. Saito et al. (2001) [64]	Non-threaded tool
A5083	FSP, 3 step-over passes	0.5-2 µm	450	1×10^{-1}	1200	T.R. McNelly (2010) [65]	Two different tools are used for overlapping FSP

Table 2.2 Reports on production of superplastic or multi-functional composites (No. 1)

Alloy	Processing method	Reinforcement	Reference	Difference or disadvantage of the study compared to our study
A6063, A6061	FSP, hardening of work surface or inside the material	Al_2O_3, WC, SiC whisker	Nishihara & Yamamura (2007) [12]	Local, non-flexible, non-homogenous microstructure
A6061	FSF, production of a locally continuous fiber Al composite	Thin metallic wire, Piano wire	K. Yamamura et al. (2011) [11]	Over-aging of Al and material softening during FSF process
A6061	FSP, local foaming of Al alloy	TiH_2	K. Yamamura et al. (2010) [141]	Heat treatment needed, over-aging and material softening
A6061	Powder metallurgy, producing superplastic aluminum composite	Si_3N_4	Mabuchi and Imai (1993) [142]	Time-consuming, lack of superplastic characteristics for superplastic forming
A6082	FSP, 4 overlapped multi-passes, composite enhancement using nano-particles	Al_2O_3	A. Shafiei-Zharghani et al. (2009) [91]	Multi-passes and surface machining needed
Al alloys	Hot rolling/ Powder metallurgy	TiN	Kojima and Ishikawa (2006) [96]	Elongation of 257% with a strain rate of 8.3×10^{-3}, complicated, time consuming
A2124	Powder metallurgy	SiC	Nieh (1984) [102]	High strain rate, refining grains throw TMP Many parameters to control

Table 2.2 Reports on production of superplastic or multi-functional composites (No. 2)

Alloy	Processing method	Reference	Achieved improvements, advantages or disadvantages
Ti-6Al-4V	SPF/DB	Hefti (2010) [113]	SPF/DB at low temperature, merits in aerospace applications
Titanium alloys	SPF/DB	Velicki (1995) [111]	Cost efficient method, light weight, strong structure
Ti2AlNb	SPF/DB	Zhihao (2016) [114]	236% elongation, strain rate of 3^{-4} s^{-1} uniform thickness
Al alloys (A7475, A5083, Zn-22Al)	SPF/DB	Hirobashi et al. (1995) [115]	A7475, A5083 reached fine particle levels, Zn-22Al did not have high bonding strength
Ceramics (ZrO_2/Al_2O_3, Mullite/ZrO_2, Al_2O_3/TiN	SPF/DB	Nagano and Wakai (1992) [116]	Investigated possibilities of applying SFP/DB for ceramics

Table 2.2 Reports on production of superplastic or multi-functional composites (No. 3)

Alloy	Processing method	Reinforcement	Reference	Achieved improvements, advantages or disadvantages
Zn-22Al	Pressure welding/ Laminated rolling	Low carbon steel	Azujima et al. (1991) [117]	Improved strength, Decreased ductility
Zn-22Al	High pressure casting/ hot extrusion	SiC	Kaneko et al. (1991) [118]	Improved tensile strength, Deformation resistance decreases during hot extrusion
Zn-22Al	High pressure casting/ hot extrusion	SiC	Kim et al. (1991) [119]	Composite is influenced by length and orientation of SiC, Composite lost superplasticity, elongation less than 100%
Zn-22Al	Molding/ Heat treatments	Wire mesh/ Steel plate	Uno et al. (1996) [120]	Composite was able to reduce creep deformation under high temperature and pressure, Composite can be used as a gasket material

Chapter 3

GRAIN REFINEMENT OF Zn–22Al SUPERPLASTIC ALLOY USING FRICTION STIR PROCESSING

3. GRAIN REFINEMENT OF Zn–22Al SUPERPLASTIC ALLOY USING FRICTION STIR PROCESSING

3.1 Introduction

In present chapter, experiments of FSP on superplastic Zn-22Al alloy is discussed. Especially, we demonstrate that friction stir processing can be used to increase the fineness of the grain structures of Zn–22Al superplastic alloy. In addition, the superplastic behaviour of friction stir processed Zn-22Al alloy at 150°C–350°C and the effect of FSP on strength anisotropy have been researched. The results of microstructural observations, hardness tests, and average grain size measurements are presented.

FSW has generated interest because of its association with friction stir processing (FSP), a new technique that employs FSW tools. FSP is currently being explored as a thermomechanical processing tool that can be used to transform a heterogeneous microstructure into a more homogenous microstructure. Mahoney emphasized that FSP can be selectively applied to a location within a conventional aluminium alloy sample to tailor its microstructure and achieve increased superplasticity. [143] Mahoney and Lynch have reported some practical applications of FSP, including the application of FSP to Al-, Cu-, Fe-, and Ni-based alloys to improve their material properties. Some of the demonstrated beneficial effects of FSP include the doubling of the strength of cast Ni–Al–bronze, the five-fold increase in the ductility of Al alloy A356, the increased fatigue life of fusion welds, the increased corrosion resistance of a Cu–Mn alloy, and the bending of a 25-mm-thick Al alloy 2519 plate to an angle of 85° at room temperature without surface cracking. [144]

In view of the above, superplasticity is associated with FSW and FSP. Superplasticity is the capability of certain metals to undergo extensive, neck-free, tensile deformation prior to fracture. Superplastic metals generally exhibit a high value of the strain rate sensitivity exponent, m > 0.33, during tensile deformation under proper conditions of temperature, which is characterized by the constitutive equation:

$$\sigma = k\dot{\varepsilon}^m \quad (3.1)$$

where σ is the true flow stress, k is a constant, and $\dot{\varepsilon}$ is the true strain rate. [145] For a superplastic metal, there is generally a maximum in the observed ductility at a specific strain rate, with significant losses in ductility as the strain rate is increased or decreased relative to the maximum. [146] These characteristics of a superplastic metal suggest that control of the process parameters for FSW or FSP is important.

Furthermore, FSP is believed to affect the grain size of the stirred material. Nishihara has reported initial results on the FSW of superplastic Zn–22Al eutectoid alloy, which demonstrate that FSW produces a fine grain structure within the joint part. [147]

Zn–22Al is a well-known superplastic material that has been widely used in different fields of studies. Zn–22Al can be obtained as sheet for thermal forming and is often useful in low-volume applications where tooling costs must be kept low. It is also used for electronic enclosures, cabinets and panels, business machine parts, and medical and other laboratory instruments and tools. [71] Zn-22Al has excellent properties needed for a seismic damper, such as high ductility and low work hardening. Tanaka investigated nano-sized Zn-Al alloy in order to develop a high-performance seismic damper capable of replacing conventional dampers. They produced a nano-crystalline microstructure by thermo-mechanical controlling process (TMCP) technology. They developed a maintenance-free seismic damper and put it into actual use in a building. [148]

In other hand, superplastic material with ultra-fined grains exhibit the high strain-rate and a low temperature superplasticity. Current goal in recent studies is the development of finer-grain materials that exhibit superplasticity at lower forming temperatures. There are different method for grain refining and improving the mechanical properties of materials which were discussed in section 2. However, few trials of FSP on Zn-22Al alloy have been conducted. Studies on controlling and improving the mechanical properties of this alloy have been conducted in our laboratory. For a superplastic metal that is tensile tested under proper temperature conditions, the observed ductility varies substantially with the strain rate. There are significant losses in the ductility as the strain rate is increased or decreased from the value at which the ductility is maximized. It is well known that the primary factor related to this behaviour is the rate of change of the flow stress with respect to the strain rate, which is usually measured and reported as *m*-value (strain rate sensitivity exponent). Higher values of m correspond to greater superplasticity. [146]

As is well known, one of the basic requirements for superplasticity is having a very fine grain. [146] It is expected that the grain refinement of Zn–22Al can be achieved using FSP, which could be of great significance in superplasticity studies.

The focus of this study is the effect of FSP on the microstructure of superplastic Zn–22Al. In particular, the effect of varying different tool process parameters of FSP on the grain size of superplastic Zn–22Al is discussed based on microstructural observation and hardness test results. The results of the present study also open a new channel for discussing different possible new applications and challenges of FSW and FSP, such as applying FSP to the improvement and development of fine grain structures for increased superplasticity or other purposes.

According to Hall-Petch relation [149, 150], strengthening materials by changing their average grain size would be possible. Grain boundaries impede dislocation movement. The number of dislocations within a grain have an effect on how easily dislocations can traverse grain boundaries and travel from grain to grain. So, dislocation movement and yield strength can be influenced by changing the grain size. In general metallic materials, the yield stress σ_y is related to the grain size d through the Hall–Petch equation as [149, 150]

$$\sigma_y = \sigma_0 + k_y d^{-1/2} \qquad (3.2)$$

where σ_0 is the friction stress and k_y is a positive constant of yielding associated with the stress required to extend dislocation activity into adjacent unyielded grains. This relationship demonstrates that the yield stress increases with decreasing grain size. In the absence of appreciable work hardening, the hardness HV of the material is proportional to the yield stress as HV = $3\sigma_y$ [151, 152] (Ashby and Jones, 1980). Eq. 1 can therefore be rewritten in terms of the hardness as [152]

$$\mathrm{HV} = H_0 + k_H d^{-1/2} \qquad (3.3)$$

where H_0 and k_H are constants associated with the hardness measurements. This study also discusses the relationship between the grain size and the hardness.

3.2 Experimental procedure

Experiments were performed on 2-mm-thick superplastic Zn–22Al eutectoid alloy. Fig. 3.1 shows the starting microstructure of the as-received fine-grained Zn–22Al with a grain size of 0.7 µm. Zn–22Al is a two-phase alloy consisting of Zn- and Al-rich grains. Table 3.1 gives the chemical composition of the material.

Table 3.1 Chemical composition of Zn–22Al [mass%]

Cu	Al	Mg	Ti	Zn
0.56	21.7	0.010	0.024	Bal.

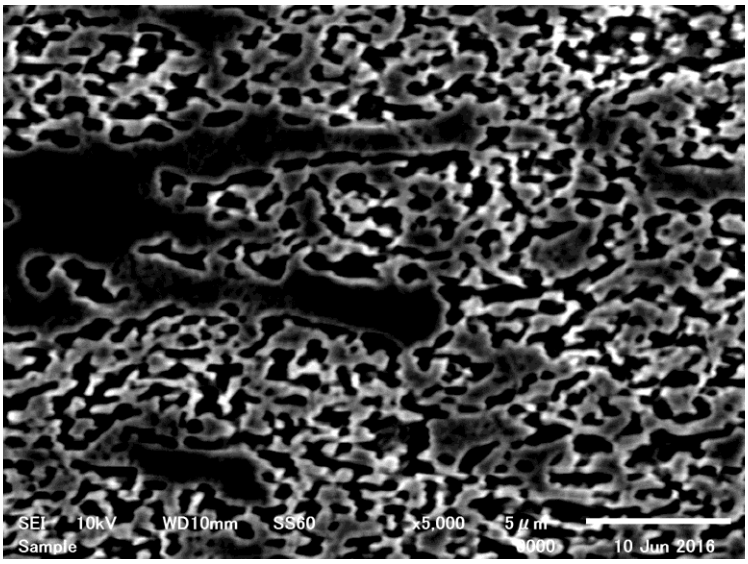

Fig. 3.1 Starting microstructure of as-received Zn–22Al

The FSP system is shown in Fig. 3.2(a). The main unit of the device consists of a vertical milling machine along with jigs, the workpiece, and a backing plate that secures these components in the desired locations. As shown in Fig. 3.2(b), the FSP tool consists of three different parts, the shank, chip, and chip cap. Fig. 3.2(c) shows the chip with its shoulder and probe labelled. The shoulder of the chip, which has a diameter of 15 mm and an angle of –5°, is made of Inconel 625 which is heat resistance, and a high-speed steel forming tap (M6 × 1) with a height of 1.5 mm was used as the probe of the chip to improve the stirring performance. It should be mention that there are many studies regarding the tool geometric effect on the process quality and how tool shape affects different properties of the material during the process [153-157]

When the tool is inserted into the material during the process, it produces plastic flow of the material in the circumferential direction due to the rotation of the tool. Moreover, a threaded probe under clockwise rotation causes the material to be drawn down by the threads along the probe surface. [157-159] The material may circulate multiple times around the tool before being deposited behind the tool. This phenomenon promotes material stirring and therefore grain refinement, void closure and oxide breakdown. [160, 161] FSP trials were conducted at various rotation and travel speeds. Table 3.2 gives the process parameters of the FSP tool.

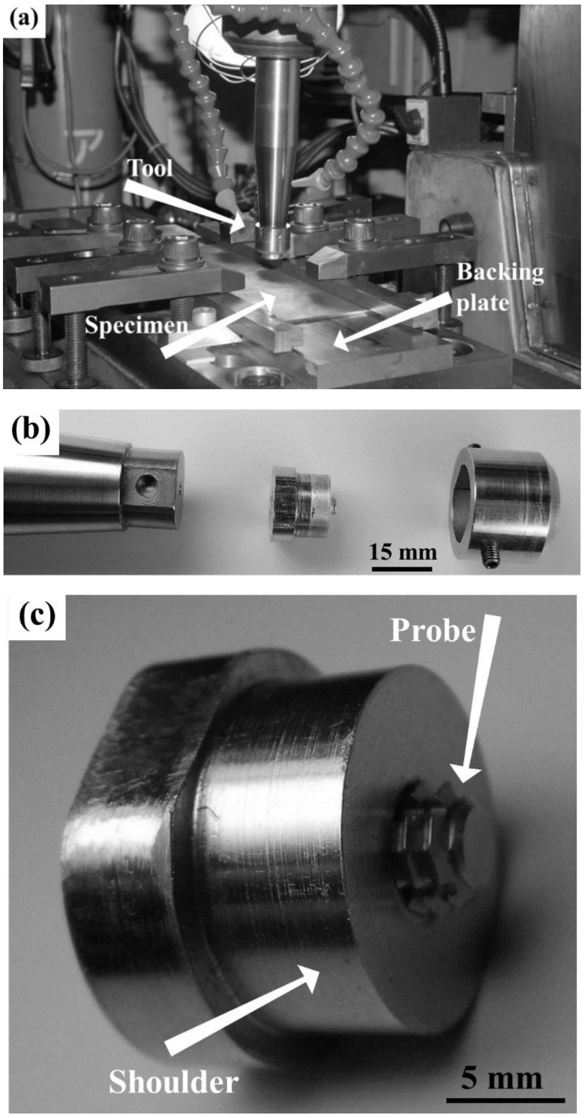

Fig. 3.2 (a) Experimental setup. (b) FSP tool parts. (c) Chip of the tool

Table 3.2 Process parameters of the FSP tool

Rotational speed [rpm]	Travel speed [mm/min]	Plunge depth [mm]	Tilt angle [°]
320–1240	100–1200	1.7	3

3.3 Results and discussions

3.3.1 Microstructural observations after friction stir processing

Fig. 3.3 and Fig. 3.4 show microphotographs of cross sections of the specimen after FSP at different travel speeds (rotational speed = 880 rpm) and rotational speeds (travel speed = 600 mm/min), respectively. The cross sections are perpendicular to the direction of processing and were etched with sodium hydroxide. The labels AS and RS in the figures indicate the advancing and retreating sides of the moving tool, respectively, and the direction of the tool rotation was counter clockwise.

It can be confirmed that the structure of the base metal was affected by FSP. Fig. 3.3 indicates that the affected area and the stir zone became smaller and narrower as the travel speed increased. Fig. 3.4 shows that the affected area spread as the rotational speed increased. The heat input for each parameter could be responsible for these results. Tang et al. have determined that at small values of the revolutionary pitch, which is defined as the ratio of the travel speed to the rotational speed, the heat input increases, allowing the temperature to increase. The revolutionary pitch decreases with decreasing travel speed and increasing rotational speed. [162] Therefore, spreading of the affected zone shown in Fig. 3.3 and Fig. 3.4 could have been caused by an increase in the heat input.

Microstructural observations within the stir zone were conducted at different sets of process parameters using a scanning electron microscope (SEM). The microstructures observed at different travel and rotational speeds are shown in Fig. 3.5 and Fig. 3.6, respectively. These images confirm that FSP drastically changed the structure of the base metal. In Fig. 3.5, although FSP changed the grain structure of the base metal, only a slight decreasing tendency in the grain size was observed as the travel speed increased. When the travel speed was increased beyond a certain limit, further increases in the travel speed had less of an effect on the grain size. Fig. 3.6 shows that at low rotational speeds, FSP had a negligible effect on the refinement of the structures of the alloy, whereas the grain size decreased as the rotational speed increased. Hardness and grain size measurements were conducted as described in the following section to confirm these results.

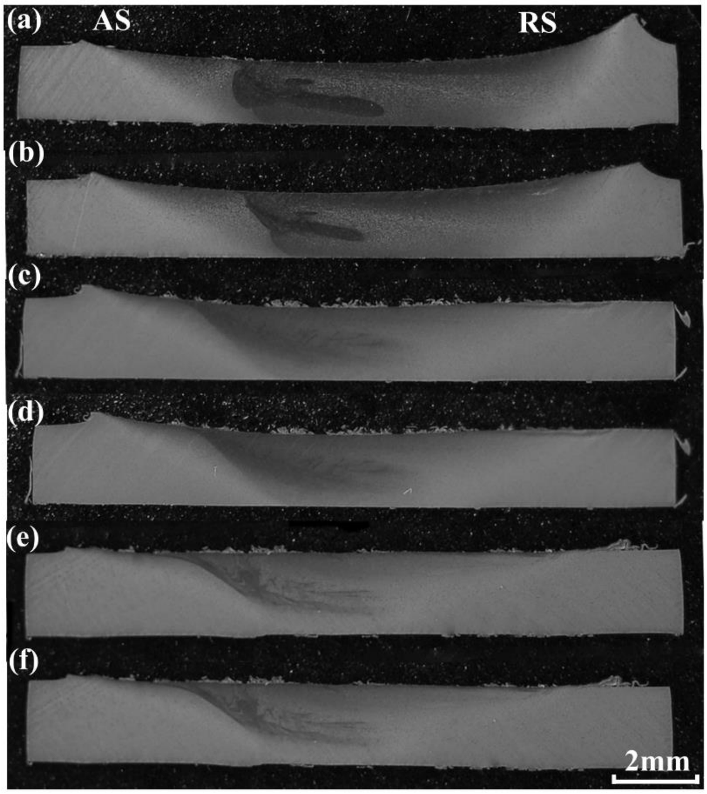

Fig. 3.3 Macrophotographs of cross sections of the specimen after FSP at a rotational speed of 880 rpm and travel speeds of (a) 100 mm/min, (b) 200 mm/min, (c) 400 mm/min, (d) 600 mm/min, (e) 800 mm/min, and (f) 1200 mm/min

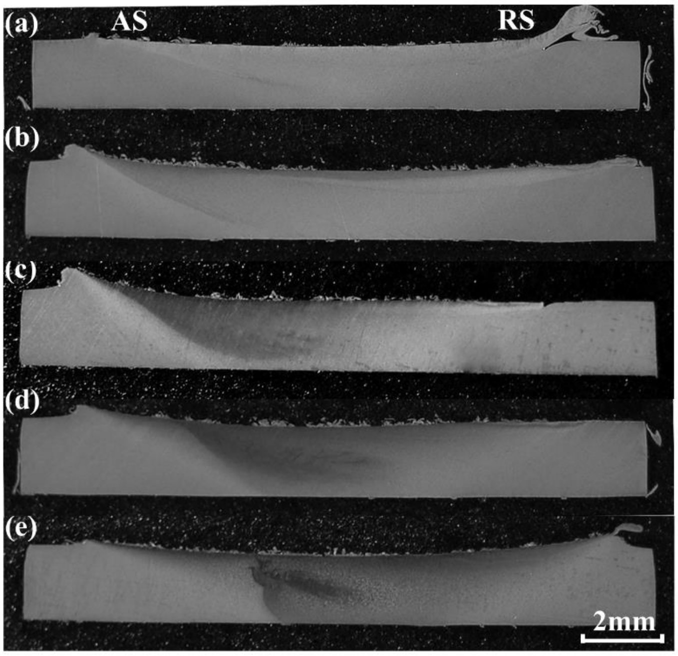

Fig. 3.4 Macrophotographs of cross sections of the specimen after FSP at a travel speed of 600 mm/min and rotational speeds of (a) 315 rpm, (b) 440 rpm, (c) 620 rpm, (d) 880 rpm, and (e) 1240 rpm

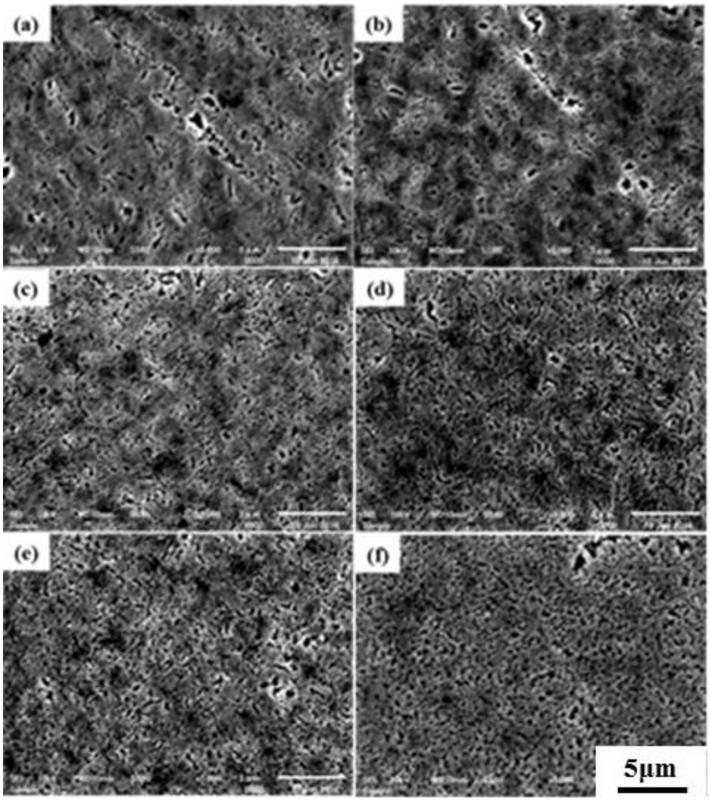

Fig. 3.5 SEM secondary electron images showing the stir zone obtained at a rotation speed of 880 rpm and travel speeds of (a) 400 mm/min, (b) 600 mm/min, and (c) 800 mm/min, (d) 1000 mm/min , (e) 1200 mm/min

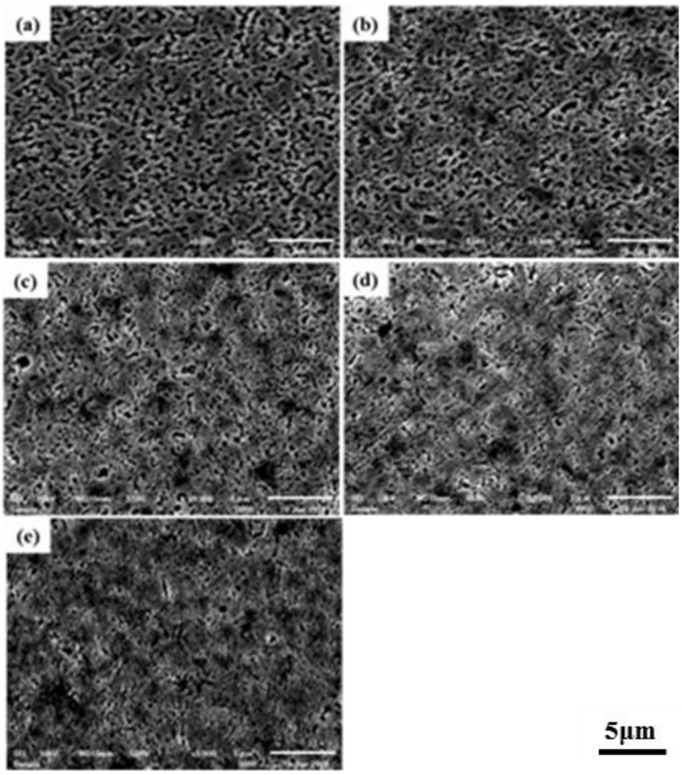

Fig. 3.6 SEM secondary electron images showing the stir zone obtained at a travel speed of 600 mm/min and rotational speeds of (a) 315 rpm, (b) 440 rpm, (c) 620 rpm, (d) 880 rpm (e) 1240 rpm

3.3.2 Hardness and average grain size measurements

The hardness of the stir zone was measured for each parameter. Fig. 3.7 shows the effect of different travel speeds on the hardness of the stir zone. The hardness within the stir zone after FSP was greater than that (107HV) of the base metal before the process under all considered sets of process parameters. However, the hardness results shown in Fig. 3.7 also indicate that the hardness does not change significantly with increasing travel speed.

Fig. 3.8 shows the relationship between the rotational speed and the hardness. The hardness of the alloy after FSP was greater than the initial hardness. Additionally, higher rotational speeds resulted in increased hardness. The reason for this hardening may be the occurrence of grain refinement during FSP.

Fig. 3.9 shows the average grain size in the stir zone after FSP at different travel speeds. Comparing these values to the average grain size of the alloy before the process, which was 0.7 μm, finer grains were obtained after FSP under all considered travel speeds. Additionally, a slight decreasing tendency in the grain size was observed as the travel speed of the tool increased up to 1200 mm/min, after which the grain size began to increase again with further increases in the travel speed. Fig. 3.10 shows the effect of the rotational speed on the average grain size. The grain size decreased as the rotational speed increased. Additionally, low rotational speeds had less of an effect on grain refinement, whereas higher rotational speeds resulted in lower grain sizes.

Comparing the hardness and grain size results reveals that hardness and grain size are inversely proportional to one another; that is, the hardness increases as the grain size decreases.

There could be many different reasons that the variation in the process parameters affects and refines the microstructure of the material. Stirring action during FSP is one of the main reasons for the obtained results. Because the probe part of the FSP tool is responsible for stirring the material, its shape and size are important. The rolled tap used as the probe part in this study had a strong beneficial effect on the stirring. The variation in the heat input with changes in the rotational and travel speed could be another reason. According to Frigaard, the friction heat created during FSP is directly related to the rotational speed [163]; the heat input increases as the rotational speed increases. Therefore, the revolutionary pitch plays an important role in the process in that a finer revolutionary pitch results in a greater frictional heat input, which changes the grain structure. Furthermore, Zn–22Al is a strain rate-dependent material, which makes it more complicated with regard to process parameters and revolutionary pitch. This study could provide accurate estimates of the appropriate process parameters to use during the FSP of superplastic alloys. In future work, a more detailed investigation of the tool size and shape would also help to provide a better understanding of how different FSP parameters affect the microstructure of the stirred material.

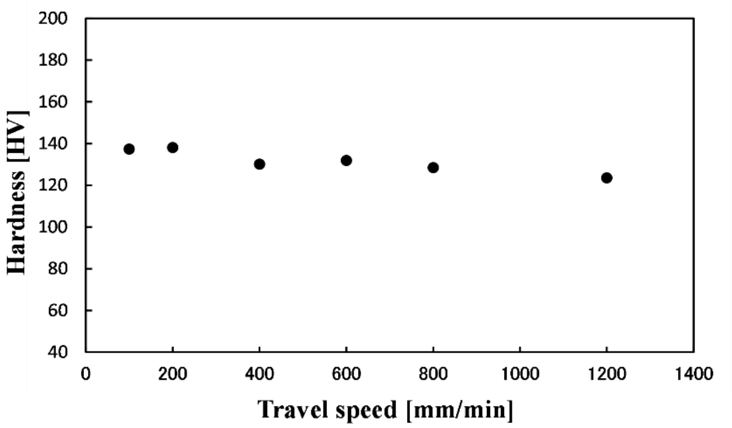

Fig. 3.7 Effect of the travel speed on the hardness of the stir zone

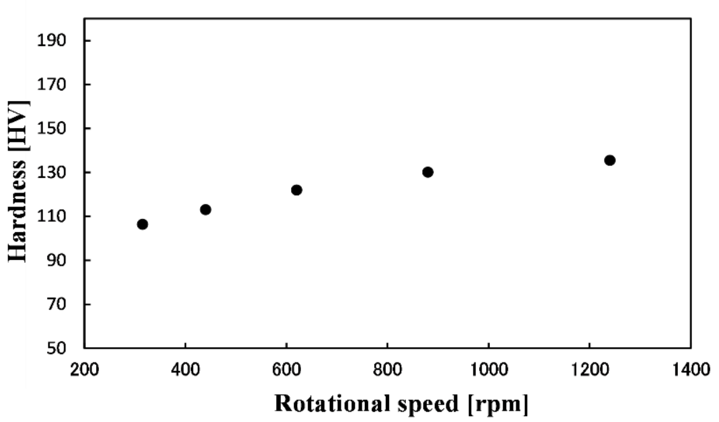

Fig. 3.8 Effect of the rotational speed on the hardness of the stir zone

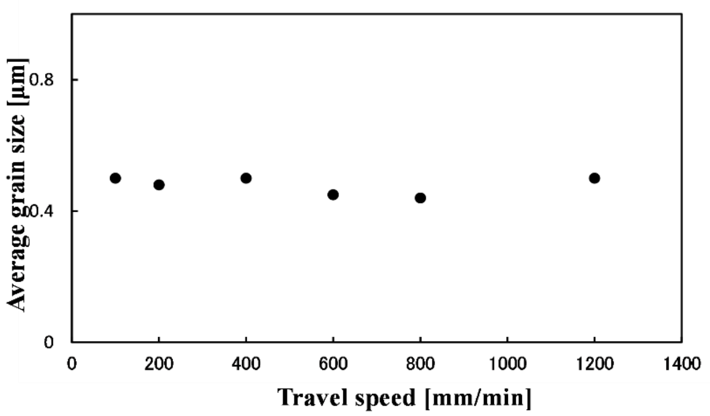

Fig. 3.9 Effect of the travel speed on the average grain size

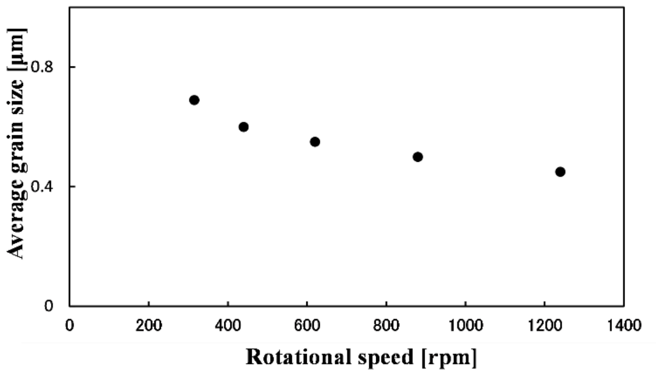

Fig. 3.10 Effect of the rotational speed on the average grain size

3.3.3 Development of fine-grained Zn–22Al plate

To develop a fine-grained Zn–22Al plate, four passes of FSP were conducted in the numerical sequence indicated in Fig. 3.11, with the space between the centers of each pair of consecutive passes set to 7.5 mm (half of the shoulder diameter). Fig. 3.11 shows a photograph of the actual friction stir-processed Zn–22Al sheet.

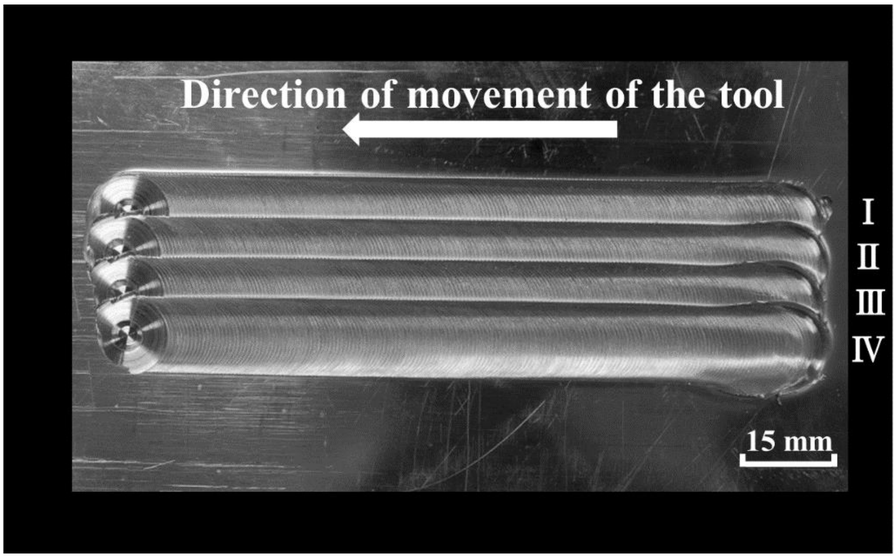

Fig. 3.11 Friction stir-processed Zn–22Al sheet after four passes (rotational speed: 880 rpm, travel speed: 600 mm/min, plunge depth: 1.7 mm with a counter clockwise rotation)

Fig. 3.12 shows a cross-sectional macrophotograph of the Zn–22Al sample after FSP. The cross section was taken perpendicular to the direction of movement of the tool. Different areas of the processed material are labelled (a)–(d). The microstructures of the unaffected zone, the heat affected zone (HAZ), the thermo-mechanically affected zone (TMAZ), and the stir zone are shown in Fig. 3.13. Microphotographs were obtained after the specimens had been etched with sodium hydroxide. FSP significantly changed the structure of the base metal. Notably finer grain structures were observed within the stir zone after FSP. The grain structure was affected in the stir zone and was finer that the grains of the base metal.

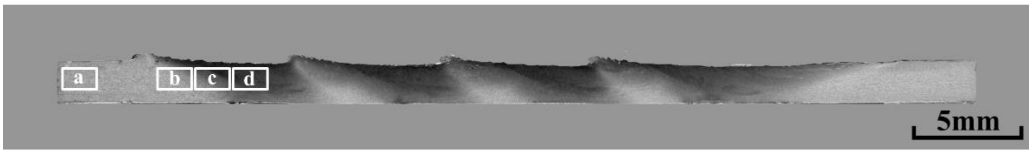

Fig. 3.12 Cross-sectional macrophotograph after FSP showing the locations of (a) the unaffected zone, (b) the HAZ, (c) the TMAZ, and (d) the stir zone

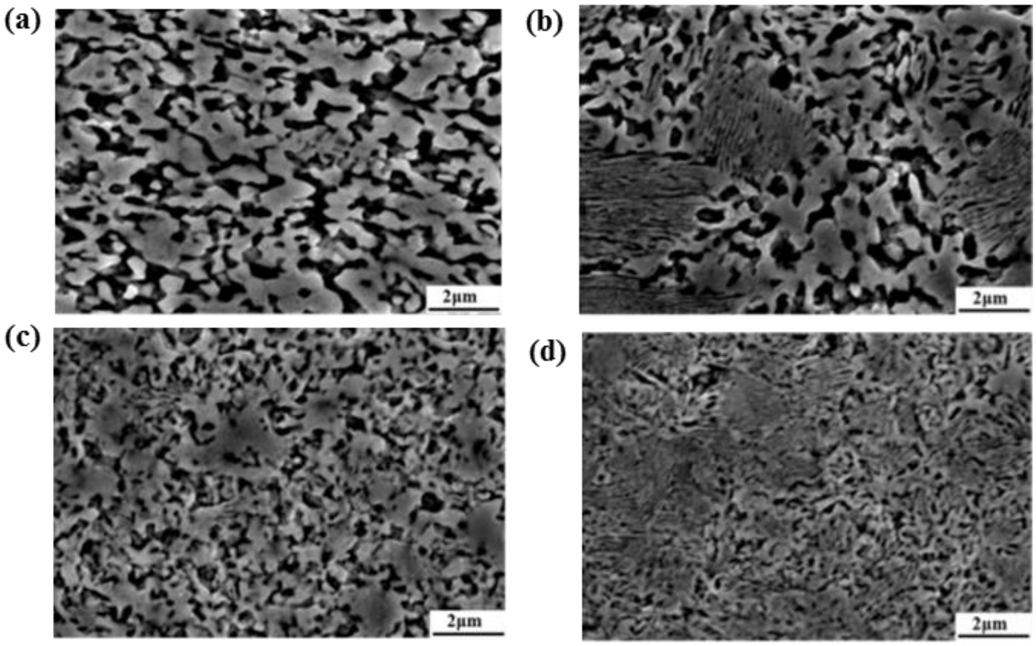

Fig. 3.13 SEM secondary electron images of (a) the unaffected zone, (b) the HAZ, (c) the TMAZ, (d) the stir zone

Fig. 3.14 shows the hardness distribution at different distances from the surface of the FSP cross section. The stir zone was considerably harder than the base metal and reached a maximum of approximately 160 HV. The hardness increased toward the stir zone, which supports the conclusion that microstructures within the stir zone are finer than those in the base metal.

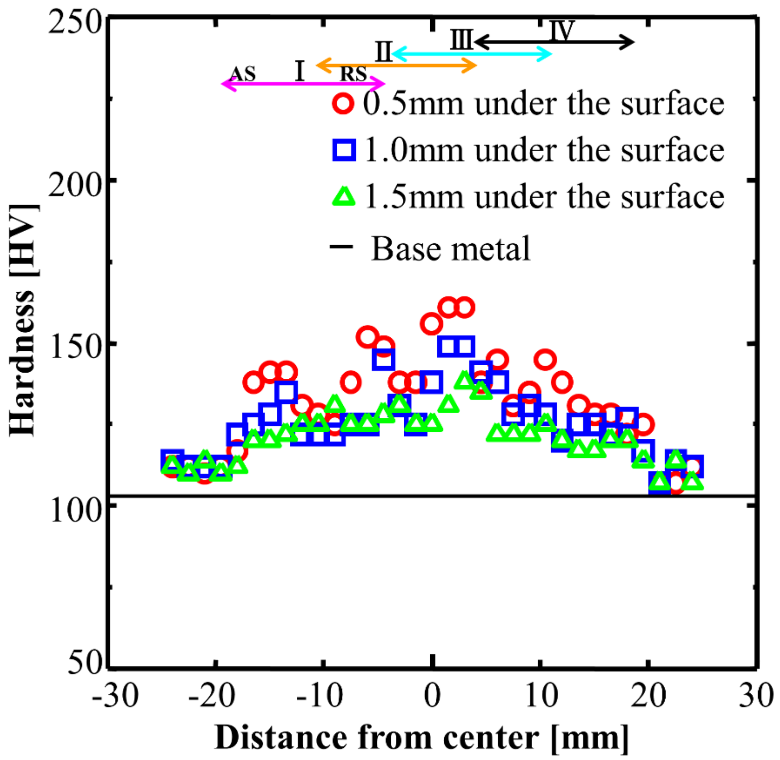

Fig. 3.14 Hardness distribution in FSP cross section.

Fig. 3.15 shows the average grain size distribution 1.0 mm under the surface of the cross section. The stir zone was found to have a considerably finer grain than the base metal. The average grain size of 0.7 μm for the base metal was reduced to 0.3 μm within the stir zone. As shown in Figs. 3.14 and 3.15, the hardness increases as the grain size decreases. In the present study, the relationship between the grain size and the hardness in different parts of the processed area (base metal toward the stir zone) are shown in Fig. 3.16. The hardness is directly proportional to the grain size, and the Hall–Petch relation of the hardness was found to hold in friction stir-processed Zn–22Al. For Zn–22Al, this relation, the constants in which were calculated using the method of least squares, is given as

$$\mathrm{HV} = 70 + 45\, d^{-1/2}. \qquad (3)$$

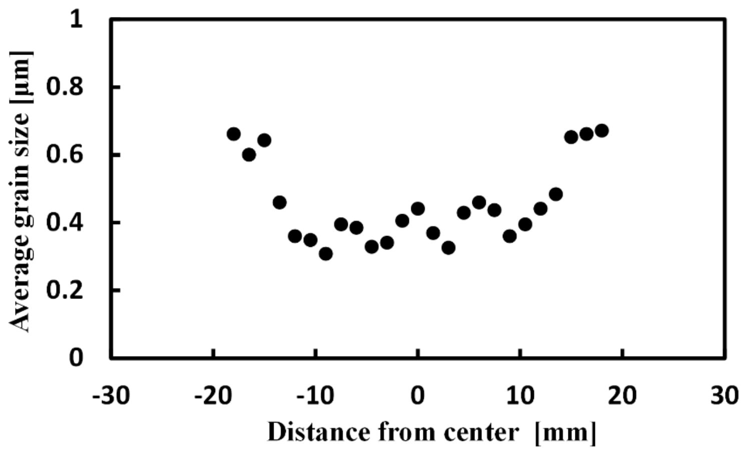

Fig. 3.15 Average grain size distribution in FSP cross section

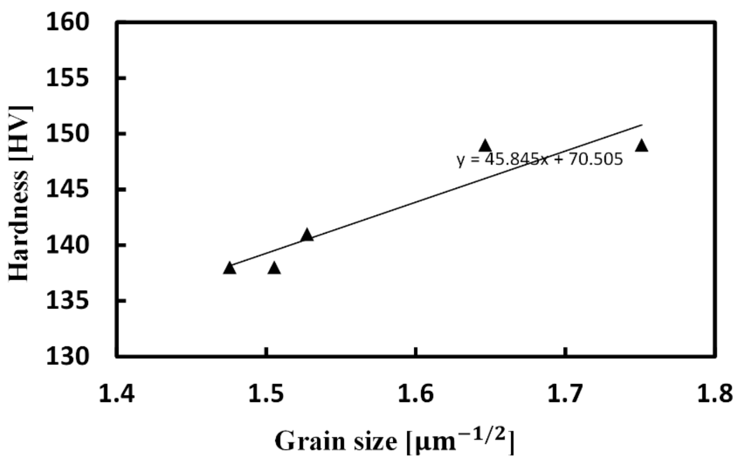

Fig. 3.16 Hall–Petch relation for friction stir processed Zn–22Al and experimental data for the hardness plotted against the inverse square root of the grain size *d*.

3.3.4 Tensile properties and superplastic characteristics

Tensile specimens were taken parallel to the direction of FSP for carrying out the tensile test. Fig 3.17 shows the effect of cross-head speed on tensile strength of Zn-22Al superplastic alloy at different angles to the rolling directions. It can be confirmed that different rolling directions cause difference in tensile strength.

It can be observed that FSP significantly decreases the effect of rolling direction on the strength of Zn-22Al alloy. Moreover, strength was increased due to the finer grain structure caused by FSP (Fig. 3.18)

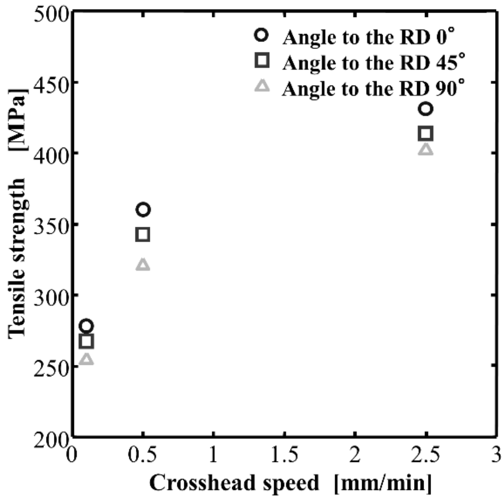

Fig. 3.17 Effect of cross head speed on tensile strength in as-received Zn-22Al. (RD: rolling direction)

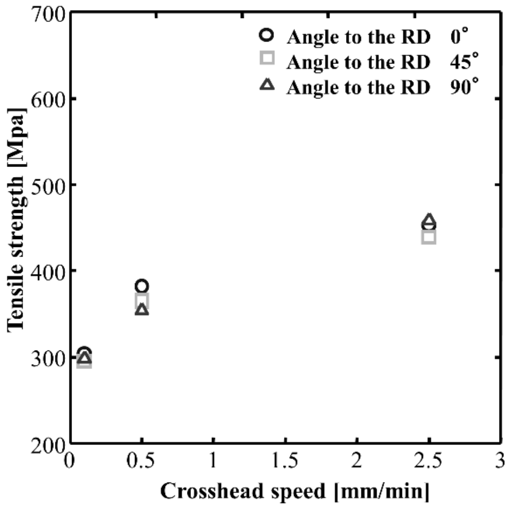

Fig. 3.18 Effect of cross head speed on tensile strength in friction stir processed Zn-22Al. (RD: rolling direction)

To investigate the superplastic behavior of friction stir processed Zn-22Al in high temperatures, an electric furnace using ceramics fiber heater was developed and attached to the tensile testing machine to provide a superplasticity tensile testing system. (Fig. 3.19)

A diameter of 0.65mm silica-glass K thermocouple was used to measure heat

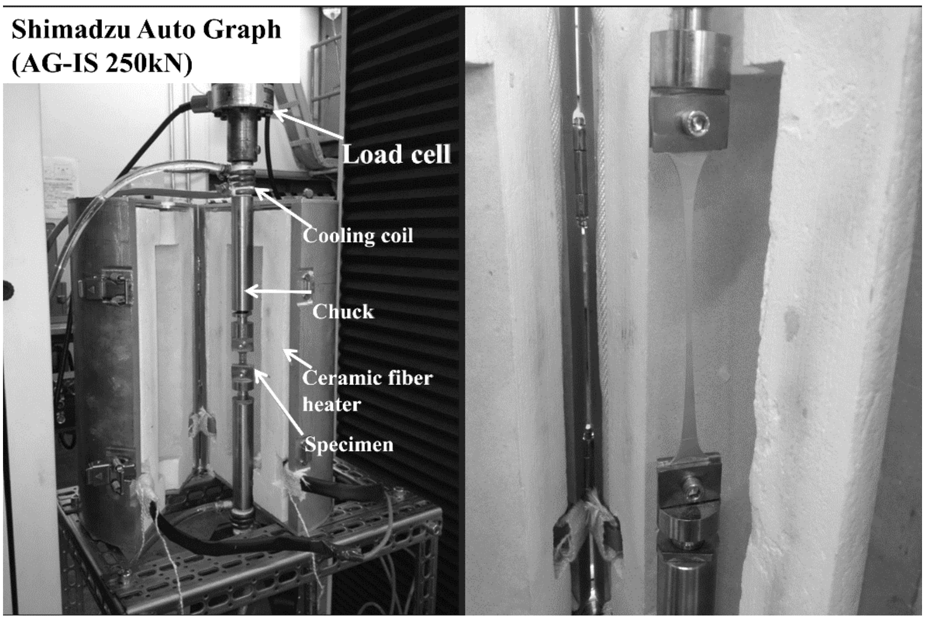

Fig. 3.19 Superplasticity testing system

during the tests.

Tensile test samples were taken parallel to direction of FSP. Tensile tests were performed at five different temperatures and seven different cross-head speeds. Effect of strain rate on tensile strength at different temperatures before and after FSP is shown in Fig. 3.20 and 3.21.

Comparing the results, a slight difference in curve of the graph for as-received and friction stir processed Zn-22Al can be confirmed. Tensile strength decreases as the temperature goes higher and it starts to increase again as the temperature reaches 350 °C in both processed and un-processed alloys.

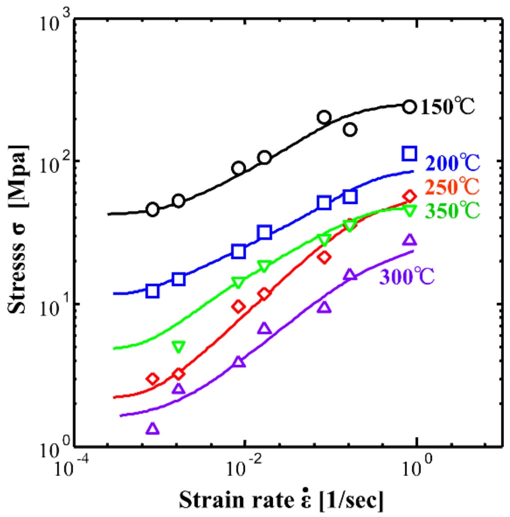

Fig. 3.20 Effect of strain rate on stress in as-received Zn-22Al

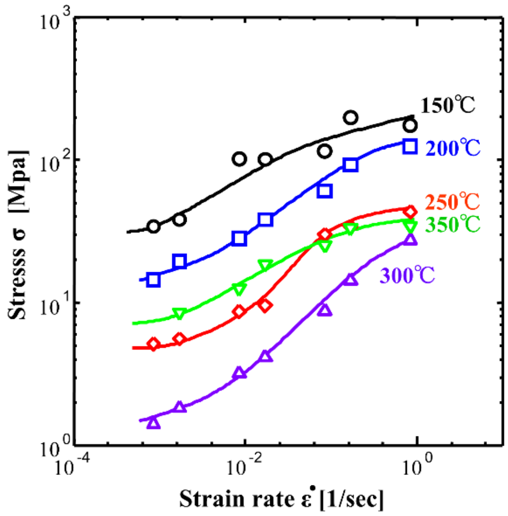

Fig. 3.21 Effect of cross head speed on tensile strength in friction stir processed Zn-22Al

Fig. 3.22 and 3.23 show the effect of strain rate on strain rate sensitivity exponent (*m*-value) for as-received and friction stir processed Zn-22Al alloy. *m*-value increases as the temperature goes higher. The highest *m*-value is obtained at 250 °C. A high *m*-value at 300 °C can still be observed but it decreases at 350 °C. Comparing Fig. 3.22 with Fig. 3.23, it can be confirmed that higher *m*-values in friction stir processed Zn-22Al alloy are achieved. Material grains have been refined after the FSP, and this could be the reason of the higher *m*-values after FSP comparing to the results for as-received alloy. Fig. 3.24 shows the tensile test photo examples at five different temperatures for as-received alloy. Increase in elongation can be observed as the temperature goes higher till 250 and 300 °C, and elongation was decreased at 350 °C.

Fig. 3.25 shows the photos of friction stir processed Zn-22Al subjected to tensile tests (Conditions and parameters are same as in Fig. 3.24 as-received alloy). Although partially a higher superplasticity can be observed, and a higher *m*-value is obtained, still a heterogeneous growth in friction stir processed samples can be seen compared to the samples of base metal. The reason could be that the grains of the material are still heterogeneous after FSP. This condition could be improved through applying multi-passes of FSP without shifting the tool, which would obviously have the disadvantage of being time consuming.

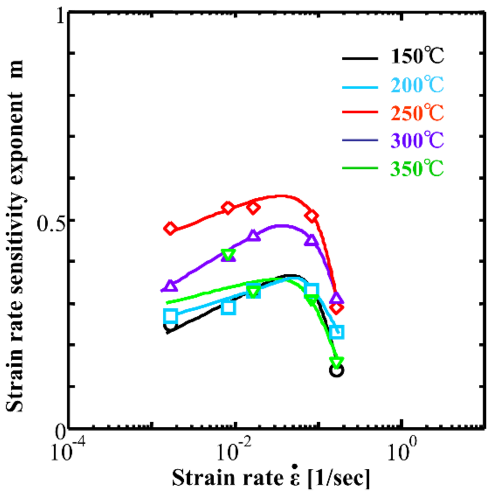

Fig. 3.22 Effect of strain rate on m-value in as-received Zn-22Al.

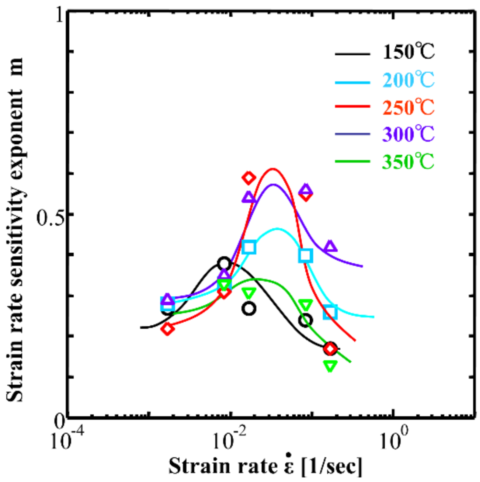

Fig. 3.23 Effect of cross head speed on m-value in friction stir processed Zn-22Al.

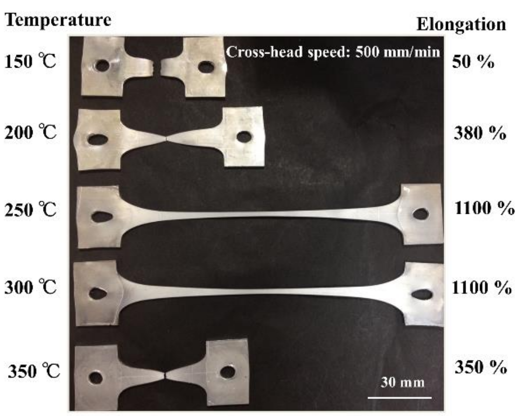

Fig. 3.24 Elongation in as-received Zn-22Al

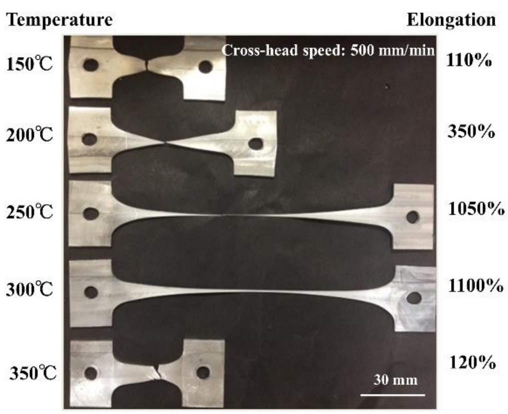

Fig. 3.25 Elongation in friction stir processed Zn-22Al

3.4 Conclusions

Improvement of the mechanical and superplastic properties of Zn-22Al alloy was demonstrated to be possible. Microstructural changes in superplastic Zn–22Al alloy caused by FSP were investigated in this study. In particular, the change in the average grain size and superplastic behaviour of Zn-22Al at temperatures 150~350 °C was experimentally determined, and the following results were obtained.

1. Grain refinement of the Zn–22Al alloy by FSP was confirmed to be possible.
2. Hardness of Zn–22Al increased toward the stir zone after FSP as a result of grain refinement.
3. Average grain size of 0.7 μm for the base metal was reduced by FSP to 0.3 μm within the stir zone.
4. Relationship between the hardness and the grain size can be accurately expressed by the Hall–Petch relation.
5. A small decreasing tendency in the grain size with increasing tool travel speed was observed, but when the travel speed was increased beyond a certain limiting value, further increases had little effect on grain refinement.
6. Higher rotational speeds resulted in finer grain size, whereas low rotational speeds had a negligible effect on the grain size.
7. Tensile strength increases after FSP. Additionally, in friction stir processed Zn–22Al, a high strain rate sensitivity exponent m of 0.59 was obtained on the high strain rate side at 250 °C.
8. Comparing the tensile testing results before and after FSP at 200 °C, it was confirmed that the flow characteristic curve clearly shifted to the high strain rate region after FSP.

Chapter 4

PRODUCTION OF A SUPERPLASTIC VIBRATION-DAMPING STEEL SHEET COMPOSITE USING FRICTION STIR FORMING

4. PRODUCTION OF A SUPERPLASTIC VIBRATION-DAMPING STEEL SHEET COMPOSITE USING FRICTION STIR FORMING

4.1 Introduction

This chapter proposes a novel method of manufacturing composite vibration-damping steel sheet with Zn-22Al superplastic alloy using friction stir forming (FSF). As it was discussed earlier, there is a need for improving mechanical properties and also compositing Zn-22Al alloy. In present study, we attempted to mechanically interlock Zn-22Al superplastic alloy to thin steel sheet and develop a vibration-damping composite alloy.

Recently, it has been shown that superplastic flow plays an important role in FSW, facilitated by deformation-induced dynamic recrystallization [164, 165]. The rotating tool stirs the contiguous metals together into the weld zone. The weld zone is the result of solid-state, superplastic processing involving dynamic recrystallization [166]. FSW has especially been generating interest because of its association with friction stir processing (FSP), a new technique that employs FSW tooling. FSP is being explored as a thermomechanical processing tool to transform a heterogeneous microstructure into a more homogenous microstructure. Mahoney et al. reported that FSP can be selectively applied to a location within a conventional aluminum alloy to tailor the microstructure for superplasticity. [143] Considering the excellent fluidity of aluminum alloy in FSW and FSP, recently a new micro-forging method has been developed and has been applied to mechanical interlocking. This process is referred to as friction stir forming (FSF). The FSF process uses the friction heat and the plastic deformation generated between a rotating tool and the material being forged. This technique has being studied in our laboratory. [9-11, 164]

By controlling the process parameters of FSF tool, Zn-22Al can reach its highest superplastic condition during the process which raises the possibility of occurring SPF/DB during friction process. Therefore, in a unique way by combining FSF and superplastic characteristic of Zn-22Al, a vibration-damping composite alloy with improved mechanical properties could be developed.

4.2 Experimental procedure

Experiments are performed on 2 mm thick Zn-22Al superplastic alloy. Table 4.1 shows the chemical composition of the material.

Table 4.1 Chemical composition of Zn-22Al [mass%]

Cu	Al	Mg	Ti	Zn
0.52	21.1	0.010	0.034	Bal.

The FSF system and FSF tool are same as our previous study which was presented in previous chapter. [167] Shoulder part of the FSF tool with a diameter of 15mm, is made of Inconel 625, while a high-speed steel forming tap (M6×1) is used as the probe part of the tool. A 0.5 mm thick perforated steel sheet is inserted between two sheets of Zn-22Al superplastic alloy. FSF is applied to the upper layer of Zn-22Al. FSF causes the upper layer to soften and flow into the hole of the steel plate. It will be demonstrated that by controlling FSF tool parameters, Zn-22Al can reach its superplastic temperature during the process which leads to joining of the two layers of Zn-22Al alloy, as well as interlocking the steel sheet in the middle. (Fig. 4.1) FSF parameters are shown in Table 4.2.

Table 4.2 Process parameters of FSF tool during the process

Rotation speed [rpm]	Travel speed [mm/min]	Plunge depth [mm]	Tilt angle [°]
440–880	25–200	1.98	3

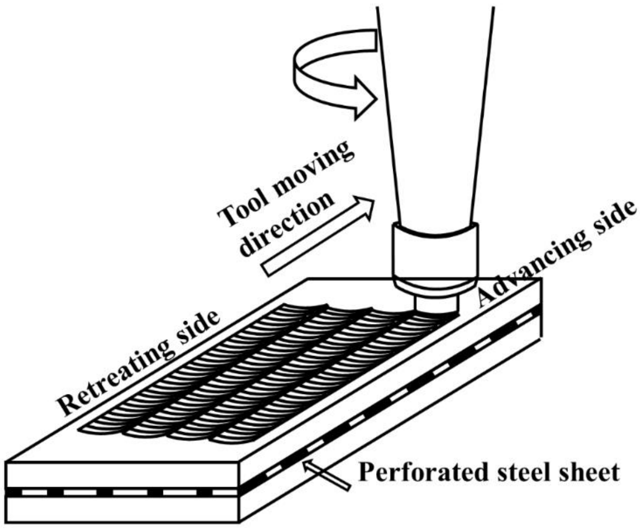

Fig. 4.1 Schematic illustration showing experimental method

4.3 Results and discussions

4.3.1 Microstructure observation

Fig. 4.2 and 4.3 show macro-photos of the cross section. The cross sections are perpendicular to the direction of processing and are etched with sodium hydroxide (black parts show steel sheet). Advancing Side (AS) and Retreating Side (RS) are same for all figures (direction of the tool rotation is counter-clockwise). As mentioned earlier in Table 4.2, Experiments are performed in various travel speeds and rotation speeds. Flow of material into the holes of steel sheet increases as the revolutionary pitch (travel speed/ rotation speed) decreases. Small revolutionary pitch also allows the temperature to increase. [168] But it can be recognized that an extremely low revolutionary pitch might cause excessive stirring and over-softening which leads to a decreased material flow. Fig. 4.2 is an example of a not proper process parameter with unjoined part between two layers of Zn-22Al. After considering temperature measurement experiments and observing different microstructures of various process parameters; it was concluded that the rotation speed of 440 rpm with a travel speed of 25 mm/min and a plunge depth of 1.98 mm are the best process parameters for this experiment as the two sheets of Zn-22Al get joined to each other (Fig. 4.3). It will be discribed later that SPF/DB and pressure welding are considered to be the two main reasons of the joining process of Zn-22Al sheets.

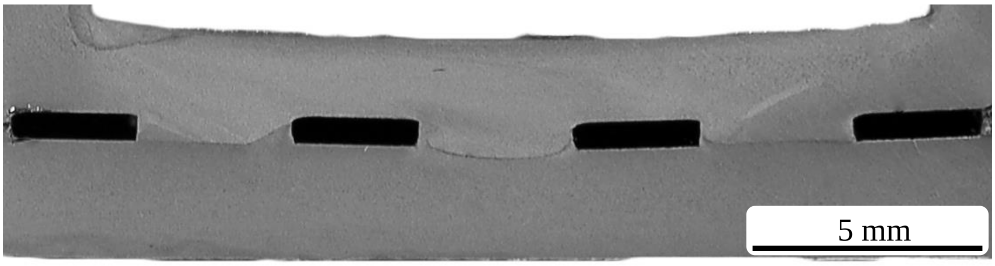

Fig. 4.2 Macro-photo of cross-section after FSF for a rotation speed of 620 rpm and a travel speed of 25 mm/min

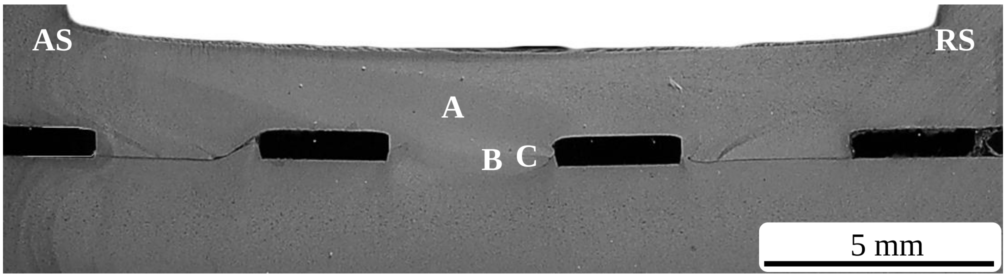

Fig. 4.3 Macro-photo of cross-section after FSF for a rotation speed of 440 rpm and a travel speed of 25 mm/min

Fig. 4.4 illustrates the base metal microstructures (starting workpiece microstructures for Zn-22Al as-received). Fig. 4.5 shows the microstructures of the stir zone. Micro-photos were obtained after etching the specimens with sodium hydroxide. FSF has significantly changed the structure of the base metal. Notably finer grain structures were obtained after FSF process within the stir zone. Average grain diameter of base metal (1.0 μm) was reduced to 0.6 μm within the stir zone due to FSF. Fig. 4.6 shows the thermo-mechanically affected zone (TMAZ) and Fig. 4.7 shows that the unjoined part between two layers of Zn-22Al has been disappeared.

It was concluded that the process parameters (rotation speed and travel speed) must be perfectly appropriate to create the proper amount of heat for a good plastic flow of material and occurrence of the superplastic phenomena of Zn-22Al. As it has been reported that superplasticity occurs during FSP beside the dynamic recrystallization [119, 120] and because the probe part of the tool does not touch the lower Zn-22Al alloy sheet, the reason that the bottom layer gets joined to the upper layer could be superplastic forming/diffusion bonding (SPF/DB). Therefore, control of temperature by controlling the process parameters would be highly important, although very difficult in FSF processes.

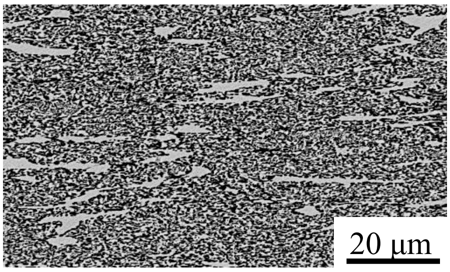

Fig. 4.4 SEM backscattered image showing microstructure of Zn-22Al as-received

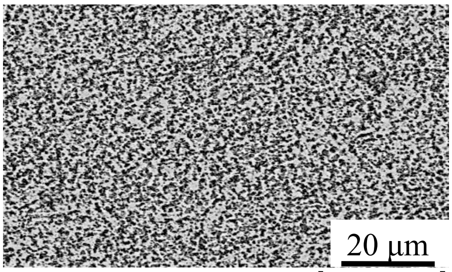

Fig. 4.5 SEM Backscattered image showing stir zone (A)

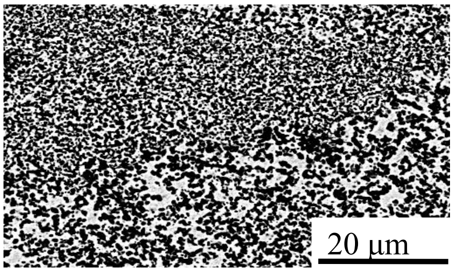

Fig. 4.6 SEM Backscattered image showing boundary between TMAZ and base metal (B)

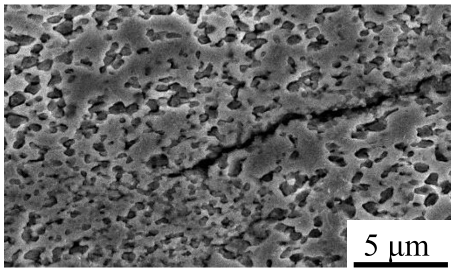

Fig. 4.7 SEM secondary electron image showing unjoined part has been disappeared (C)

4.3.2 EDS analysis and temperature measurements

Using energy-dispersive spectroscopy (EDS), line analysis were conducted to analyze the interface between Zn-22Al sheets. It can be confirmed that line analysis where the interface was expected to be disappeared shows a continuous result, therefore, it was concluded that the interface has been completely disappeared.

The thin gap between steel sheet and Zn-22Al along with EDS results shown in Fig. 4.9, indicate that there have been no metallurgical joining between two alloys.

A new discovery during electron microscope analysis was that EDS line analysis results showed an unexpected peak of Al in the interface between steel and Zn-22Al alloy (Fig. 4.9, red line). EDS mapping analysis was also carried out to confirm and explain the appearance of aluminum (Fig.4.10). It was found out that there is a thin layer of aluminum formed in the interface between steel and Zn-22Al alloy (Fig. 4.10 left bottom). Since the tool does not touch the steel layer, it was concluded that an oxide layer forms in the surface of Zn-22Al. Despite the fact that the relatively low temperature reached during FSF process will restrict intermetallic compound formation and reaction between Fe and Al atoms, there could be a possibility that through flowing of the softened material and also existence of oxygen, Al atoms have reacted with oxygen and formed an alumina layer. However, more detailed studies are needed to support this statement.

Temperature was measured in different points shown in Fig. 4.10 (K thermocouple with a diameter of 0.1 mm was used in this experiment). Temperature in point B of Fig. 4.10 (the temperature exactly under the probe) was 300°C at the maximum. The temperature in point A is the temperature for the top of the bottom layer of Zn-22Al superplastic alloy which was about 270 °C at the maximum. This temperature is close to the temperature of having the highest *m*-value (strain rate sensitivity index) and superplasticity for this alloy. [122] The outstanding characteristic of Zn-22Al to reach its highest superplasticity at a relevant low temperature, makes the occurrence of SPF/DB in area “A” very possible. By applying proper rotation and travel speed, accurate temperature of superplastic phenomena of Zn-22Al can be reached, therefore, it can be concluded that SPF/DB could be the possible reason that sheets of Zn-22Al alloy are joined together. it should be noted that the suggested method would only be possible if both upper and bottom sheets are same Zn-22Al superplastic alloys, since the superplastic characteristic of Zn-22Al alloy would let us to apply this method for producing composites which will not be possible by conventional methods. Applying the same technique for dissimilar alloys would probably not be possible and would need additional studies and experiments.

Temperature at point C exactly above the steel sheet was 280 °C at the maximum. Temperatures at points D and E decrease as we move toward the advancing side (AS) and retreating side (RS) (180 °C for point D and 170 °C for point E).

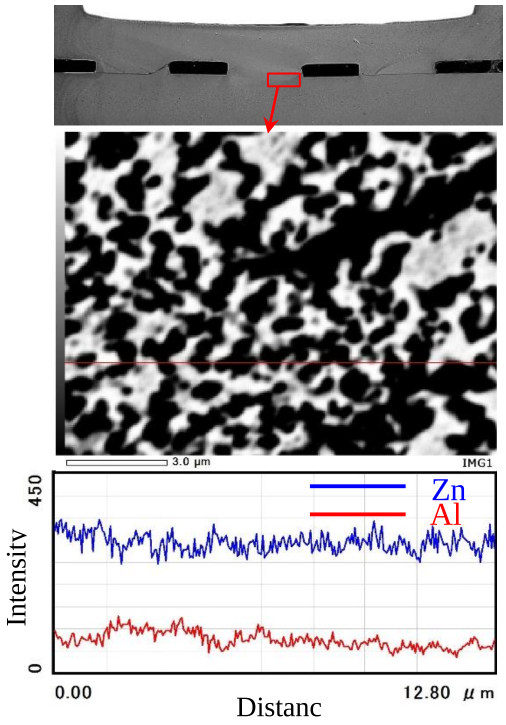

Fig. 4.8 Line analysis carried out by SEM on an etched specimen after FSF, confirms how un-joined part has been disappeared

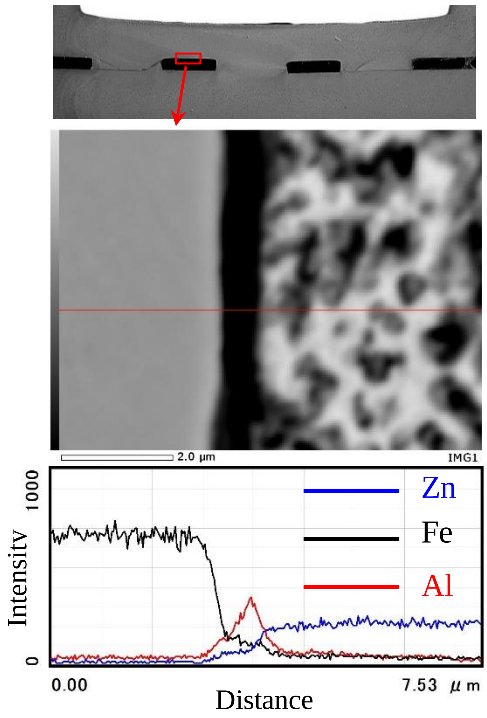

Fig. 4.9 SEM line analysis between Zn-22Al and steel sheet on a polished specimen, showing gap between two sheets

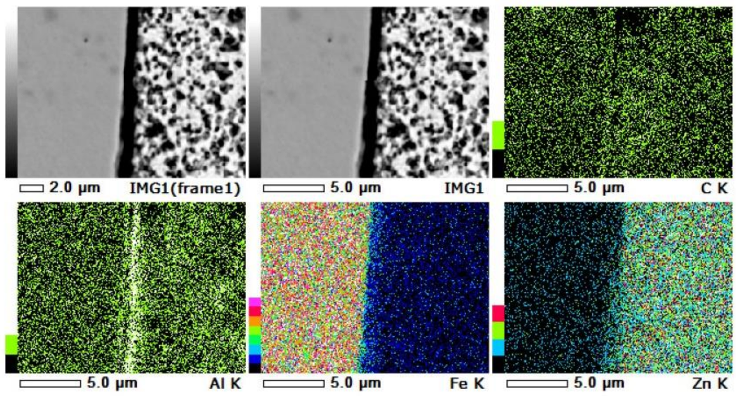

Fig. 4.10 Map analysis of interface area between steel and Zn-22Al sheet, showing built up of a thin aluminium layer (white layer in the left bottom photo)

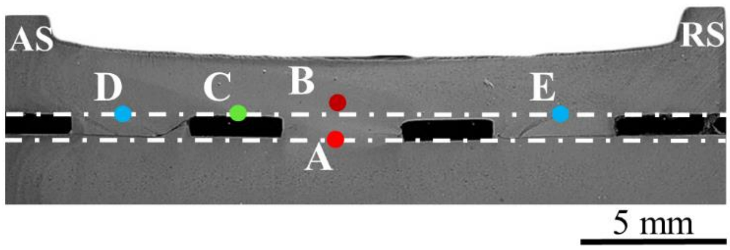

Fig. 4.10 Temperature was measured in A~D points during the FSF process with rotation speed of 440 rpm, travel speed of 25 mm/min, with a counter clockwise tool rotation

4.3.3 Developing superplastic damping plate

To develop a superplastic vibration damping steel sheet composite, FSF was carried out in six passes each overlapped two times at 5.0 mm apart (Fig. 4.11). Fig. 4.12 shows the macrostructure of the cross-section after six passes. By overlapping passes while performing the FSF, unjoined parts are eliminated. Developed superplastic vibration-damping steel sheet composite is shown in Fig. 4.13.

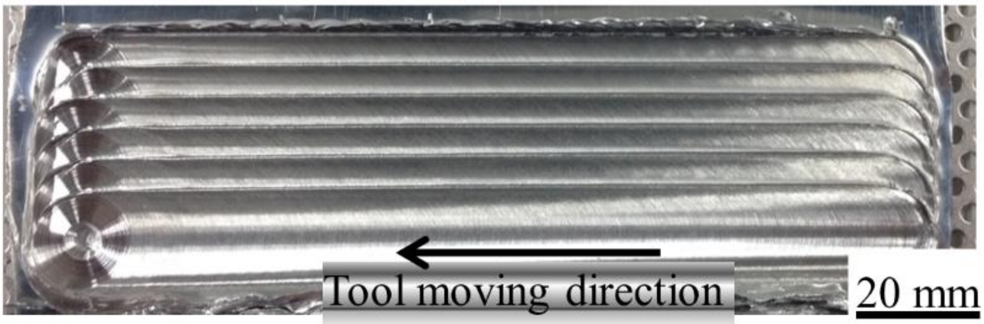

Fig. 4.11 Appearance of friction stir formed Zn-22Al with steel sheet after six passes each overlapped two times with a rotation speed of 440rpm and a travel speed of 25mm/min

Damping capacity of the developed material was measured experimentally according to JIS G 0602 (test method for vibration-damping property in laminated damping steel sheets of constrained type). Damping capacity is the ability of a material to absorb energy by converting mechanical energy into heat. A material with a good damping capacity is able to absorb mechanical vibrations or damp them out quickly, which is an important property in applications such as crankshafts and machinery bases. Damping can be expressed through different parameters such as damping ratio or the loss factor. The loss factor was calculated from the obtained displacement data (displacement of vibration was measured using a non-contact displacement meter sensor). The loss factors of Zn-22Al superplastic alloy, aluminum and steel sheet were measured as well. The results are shown in Table 4.3. It can be confirmed that the loss factor of superplastic vibration-damping steel sheet composite is the highest; therefore, it could be concluded that the developed composite is a high quality vibration-damping sheet with improved mechanical properties and a high damping capacity.

Fig. 4.12 Macrostructure of cross section after 6 passes of FSF each overlapped two times

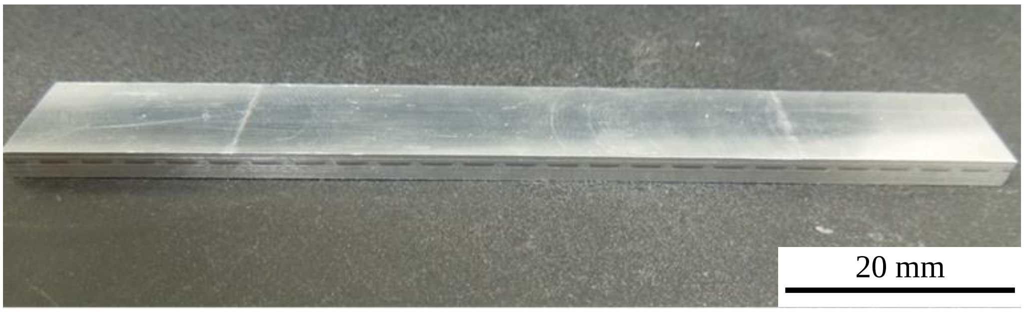

Fig. 4.13 Developed superplastic vibration-damping steel sheet composite

Table 4.3 Obtained loss factor values for different materials

	Steel sheet	Aluminum (6061)	Zn-22Al	Superplastic vibration-damping sheet
Loss factor	0.11	0.12	0.19	0.32

4.4 Conclusions

A novel method of manufacturing superplastic alloy by combining FSF and superplastic phenomena of Zn-22Al was proposed and following results were obtained:

1. It was demonstrated that by controlling the process parameters of FSP tool, Zn-22Al can reached its highest superplastic temperature, and as a result; successful join of Zn-22Al sheets and interlocking steel sheet between, would become possible.
2. It was demonstrated that FSF improves the mechanical properties of the manufactured superplastic composite while still keeping the superplastic and damping properties of the alloy. This can suggest new applications of this fabricated superplastic composite and have an impact in opening new possibilities in industrial application.
3. It was confirmed that the damping ability of superplastic vibration-damping steel sheet composite was 1.7 times bigger than the damping ability of Zn-22Al, a well-known damping alloy.

Chapter 5

FRICTION STIR FORMING FOR MECHANICAL INTERLOCKING OF INSULATED COPPER WIRE AND Zn-22Al SUPERPLASTIC ALLOY

5. FRICTION STIR FORMING FOR MECHANICAL INTERLOCKING OF INSULATED COPPER WIRE AND Zn-22Al SUPERPLASTIC ALLOY

5.1 Introduction

In present chapter, a novel method of friction stir forming (FSF) was conducted for mechanical interlocking of Zn-22Al superplastic alloy and thin copper wire insulated by polyimide. The potential development of a composite material capable of transmitting electrical energy or electric signals was studied experimentally, and it was concluded that FSF can successfully interlock insulated copper wire with Zn-22Al superplastic alloy. Same phenomena of the possibility of occurrence of SPF/DB during the process as it was described in previous chapter which combines with pressure welding to join Zn-22Al sheets is suggested. Trials of FSF were carried out on a modified vertical milling machine. The results are discussed in terms of microstructure observations, hardness distributions, and temperature measurements.

Due to the fluidity of aluminum alloys in FSW, a new micro-forging method has been developed and applied to mechanical interlocking. This process is referred to as friction stir forming (FSF). The FSF process uses the friction heat and the plastic deformation generated between a rotating tool and the material being forged.

Present study experimentally investigated the mechanical interlocking of Zn-22Al superplastic alloy to a thin insulated copper wire and the development of a new composite alloy. Zn-22Al can be obtained as sheet for thermal forming and is often useful in low-volume applications where tooling costs must be kept low. Zn-22Al is also used for electronic enclosures, cabinets and panels, business machine parts, and medical and other laboratory tools [71]. Using friction stir technique for interlocking insulated copper wire, a multi-functional composite material was developed. The insulated airtight interlock structure expands the potential of the composite material in both mechanical and electrical engineering applications.

The developed composite material is expected to have various uses, such as transmitting electrical energy or sending electric signals, and could even be applied in hermetic seal technologies. Different types of hermetic seals typically include air-resistant containers made of different materials for different purposes. Hermetic sealing has various uses, such as implantable medical devices, optical devices, electrical or electronic parts, integrated circuits (ICs), and MEMS applications [169-171]. In the past, many studies have made proposals regarding fabrication of a hermetic seal [172-175]. However, transmitting electrical or optical signals in or out of a package that has been hermetically sealed has been a difficult challenge for many companies in the electronics industry, since electrical current and light are required to enter and exit the hermetic package.

By using the FSF technique, it is expected that insulated copper wire can mechanically and hermetically be joined to Zn-22Al superplastic alloy with the capability of sending electricity or any other needed signals. This also shows the potential of a micro-forming application of FSF.

Furthermore, the knowledge of the processed zone temperature in FSW is of great interest because it determines the microstructural evolution and mechanical properties of the processed zone. The relation between temperature and quality of the processed zone has been reported in several past studies [176-178]. So far, however, only a few investigations have shown the set point temperature of the control system necessary to obtain a high-quality process, especially for superplastic materials. Some studies have suggested that superplasticity occurs during FSW [179]. Therefore, in the present study, temperature measurements were carried out to give the readers an indication of the actual temperatures during the FSF process.

5.2 Experimental procedure

The FSF system and FSF tool are the same ones used in our previous studies which were described in previous chapters. [180] FSF system is shown in Fig. 5.1. The shoulder part of the FSF tool with a diameter of 15 mm and an angle of -5° is made of Inconel 625 because of its heat resistance, and a high-speed steel forming tap (M6×1) with a height of 1.5 mm is used as the probe part of the tool to improve the stirring performance. Experiments are performed on 2 mm thick Zn-22Al eutectoid superplastic alloy. The initial microstructure of the superplastic alloy is shown in Fig. 5.2. Table 5.1 shows the chemical composition of the material (same material used in previous experiments).

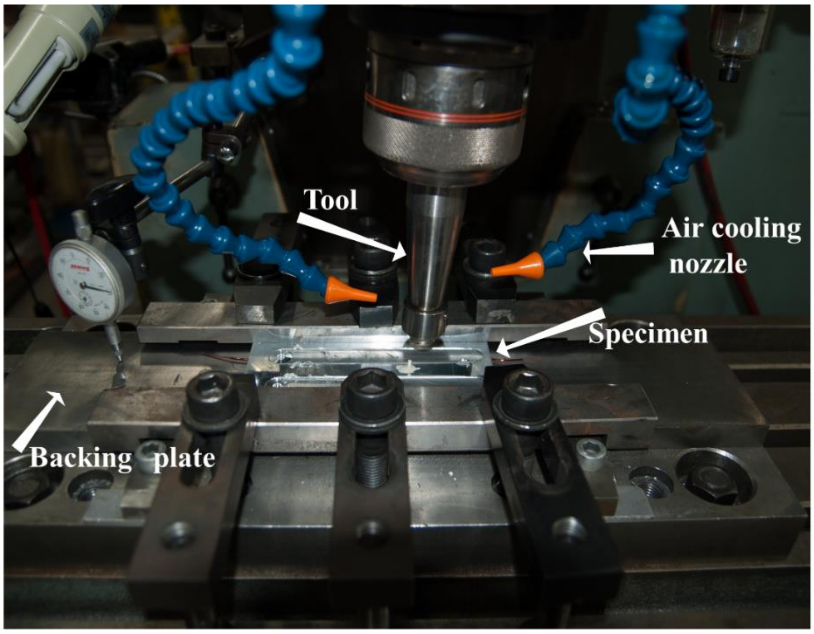

Fig. 5.1 Experimental setup for FSF experiments

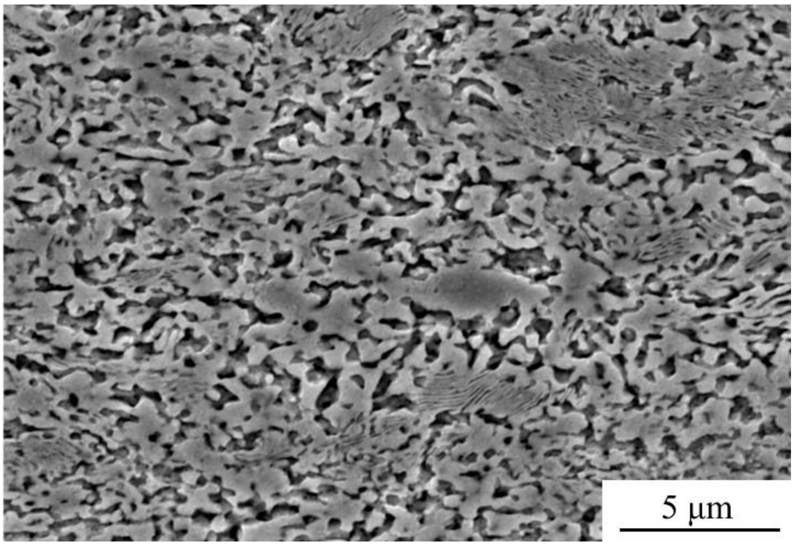

Fig. 5.2 Initial microstructure of the
as-received Zn-22Al alloy

Table 5.1 Chemical composition of Zn-22Al

Cu	Al	Mg	Ti	Zn
0.52	21.1	0.010	0.034	Bal.

Table 5.2 Process parameters of FSF tool

Rotation speed [rpm]	Travel speed [mm/min]	Plunge depth [mm]	Tilt angle [°]
315–880	25–400	1.7	3

The workpiece temperature during FSF was also measured by using an array of type K thermocouples (0.1 mm in diameter) inserted directly into the workpiece at various distances from the process centre and 2 mm under the surface. Fig. 5.3 shows the arrangement of thermocouples in a workpiece used in the experiments.

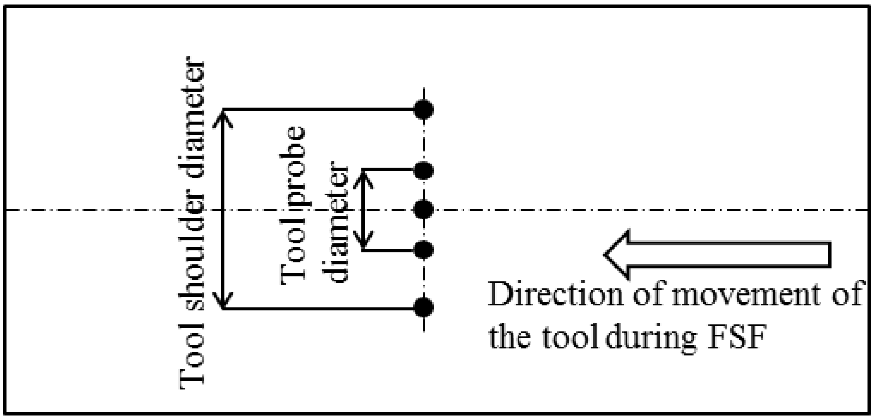

Fig. 5.3 Schematic illustration of the locations of thermocouples in the workpiece

As shown in Fig. 5.4, a guide slit is cut into Zn-22Al plate by machining, and a thin copper wire of diameter 0.5 mm, insulated by polyimide is put into the guide slit. The depth of the guide slit is 0.6 mm and the width is 0.7 mm. Another Zn-22Al plate with the same thickness is placed on top of the first plate. Then, FSF is applied to the upper Zn-22Al sheet. To perform FSF, a rotating tool with a profiled probe is plunged into and fed along the line of the guide slit. The tool rotation is counter clockwise and the plunge depth is set to 1.7 mm. The rotating tool is traversed at a 3° forward angle through the slit line. As the tool moves, friction stirring occurs and the workpiece flows into the guide slit as a result of plastic flow. This flow joins the two sheets of Zn-22Al alloy and mechanically interlocks the copper wire in the middle. Choosing the proper FSF parameters plays an important role for achieving the desired results. Table 5.2 shows the process parameters of the FSF tool during the process.

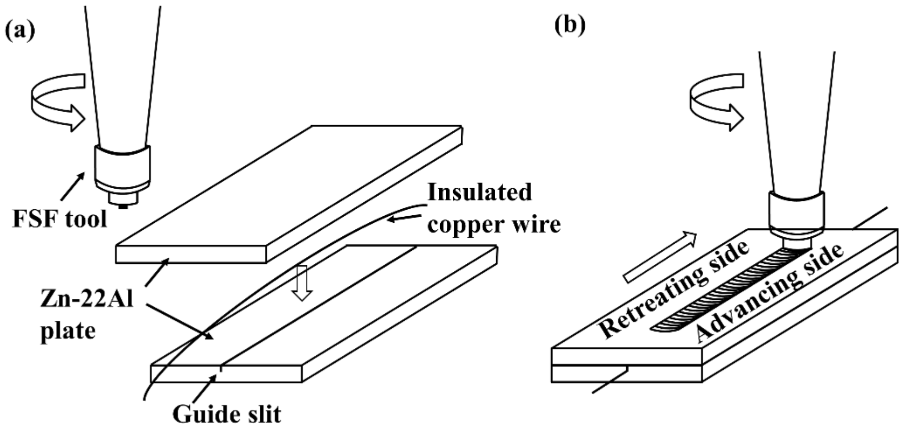

Fig. 5.4 (a) Insulated copper wire of diameter 0.5 mm is put into a slit on a Zn-22Al alloy plate, and another plate of Zn-22Al alloy is placed on top. (b) Rotating tool is plunged into and fed along the slit line to perform FSF

5.3 Results and discussions

5.3.1 Interlocking of insulated copper wire

Fig. 5.5 shows cross-sectional photos of the mechanical interlock formed between the wire and the Zn-22Al alloy. The cross sections are perpendicular to the direction of processing and are etched with sodium hydroxide.

It can be confirmed that the Zn-22Al superplastic alloy flows and completely fills the slit. The flow of the material increases as the revolutionary pitch (travel speed/rotation speed) decreases. A small revolutionary pitch also allows the temperature to increase [181].

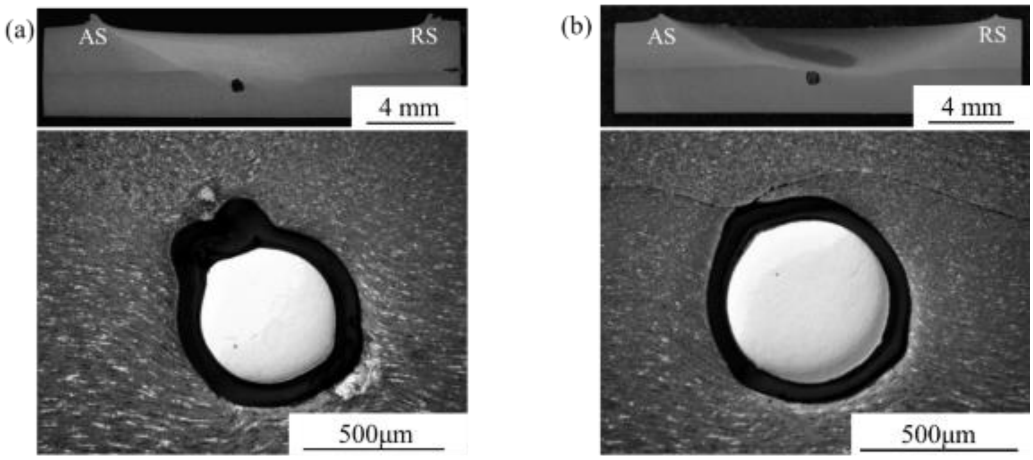

Fig. 5.5 Cross-sectional photos of mechanically interlocked insulated copper wire formed by FSF for different process parameters: (a) rotation speed of 440 rpm, travel speed of 50 mm/min (b) rotation speed of 880, travel speed of 100 mm/min. (AS: advancing side, RS retreating side)

It is also confirmed from Fig. 5.5 (a) that the interface between the two layers of Zn-22Al alloy has disappeared. Figure 5.5 (b) is an example of a non-appropriate process parameter with an unjoined interface between the two layers of Zn-22Al.

It is known that the processed material undergoes severe plastic deformation during friction stirring [182, 183]. Considering that probe part of the tool does not touch the bottom layer, the most realistic explanation of the joining of the top and bottom layers of Zn-22Al alloy, as discussed in previous studies, is the pressure welding during FSF or possibly superplastic forming and diffusion bonding (SPF/DB) [184]. Previous studies have shown that superplasticity occurs during FSW [134], but a more detailed study of superplasticity is required for proving SPF/DB in this study. A future challenge is to examine detailed data for proving SPF/DB.

SPF/DB technology of sheet materials is an advanced method of sandwich structure manufacturing that is widely used in aerospace and ship building industries. Titanium-based alloys are one of the promising candidates for applications in advanced aerospace programs because of their high-temperature strength, good fatigue life, and excellent corrosion resistance [185-189]. SPF/DB technology of titanium alloy is a well-established process for the manufacture of aircraft parts because of its advantages of weight and cost savings [190]. However, SPF/DB has some disadvantages, such as high energy consumption, tooling damage, and expensive dies and equipment. As mentioned before, the present study shows us the possibility of using the FSF technique to overcome some of the disadvantages by lowering the cost of the process and creating a fine-grain structure in the stir zone area to improve the quality of SPF/DB.

5.3.2 Microstructure observations after FSF

Fig. 5.6 shows the microstructure of the cross section. It is confirmed that FSF significantly changes the structure of the base metal. Microstructures of the base metal, heat-affected zone (HAZ), thermo-mechanically affected zone (TMAZ), and stir zone are shown in Fig. 5.7. Notably finer grain structures after the FSF process are obtained within the stir zone.

Microstructure observations of the interface between two sheets of Zn-22Al alloy in different locations were carried out. The observed locations are indicated in Fig. 5.8. It can be confirmed from Fig. 5.9 that the interface becomes thinner and disappears in area (a), which is closer to the probe and the stirring part.

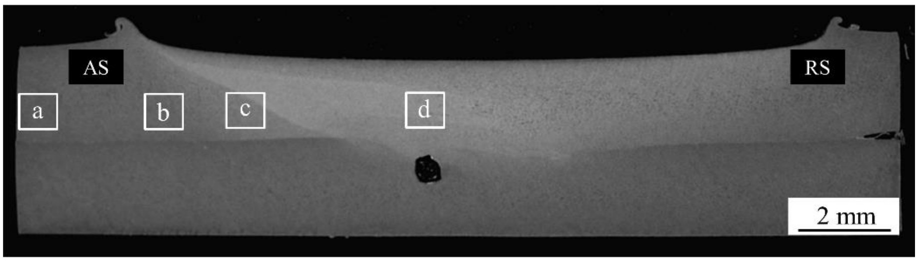

Fig. 5.6 Macro-photo of cross section after FSF for a rotation speed of 440 rpm and a travel speed of 50 mm/min

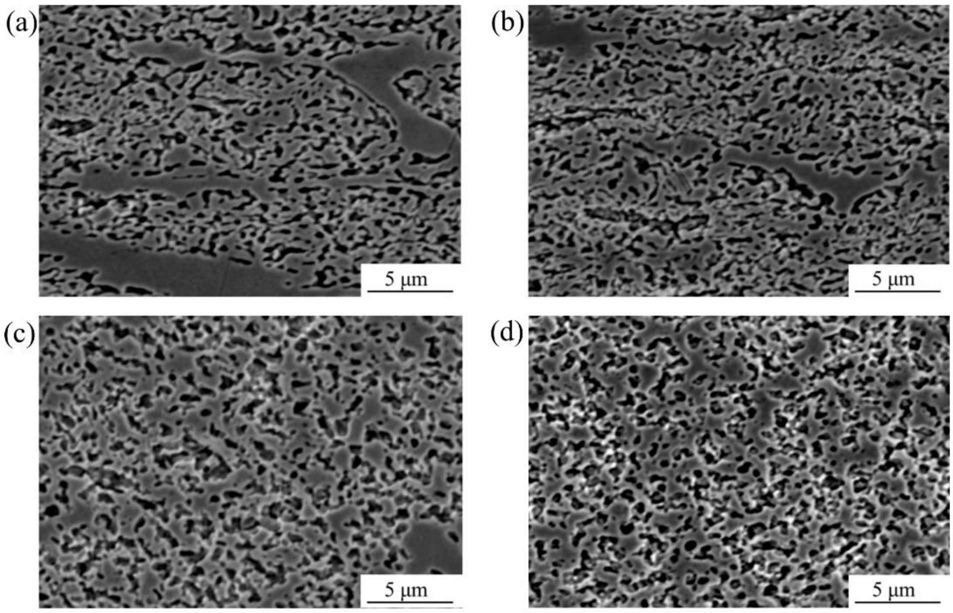

Fig. 5.7 SEM secondary electron images of cross section after FSF for a rotation speed of 440 rpm and a travel speed of 50 mm/min: (a) base metal, (b) heat-affected zone, (c) thermo-mechanically affected zone, and (d) stir zone

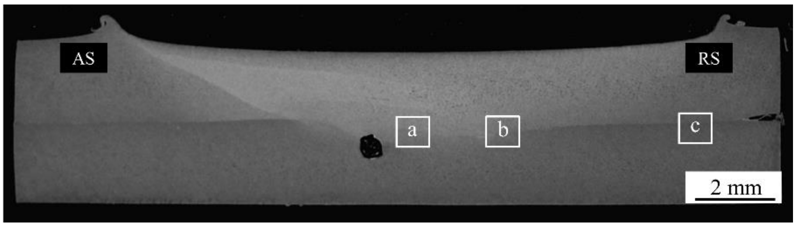

Fig. 5.8 Macro-photos of cross section after FSF for a rotation speed of 440 rpm and a travel speed of 50 mm/min with specified locations where the microstructural photos were taken

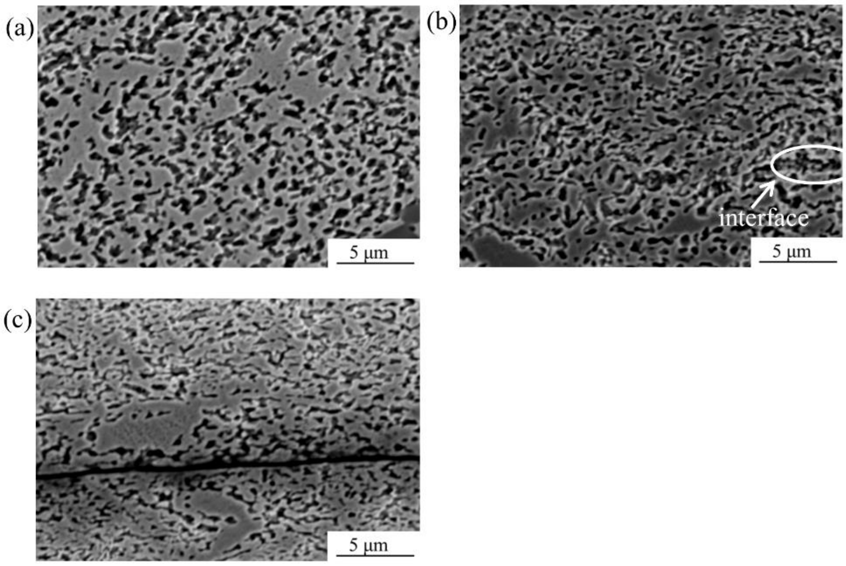

Fig. 5.9 SEM secondary electron images: (a) 2 mm under surface 1.5 mm away from the probe centre line with no unjoined interface, (b) 2 mm under surface 3 mm away from probe centre line where interface has disappeared, and (c) 2 mm under surface 7.5 mm away from the probe centre line where the interface between the two sheets can be observed

5.3.3 EDS analysis

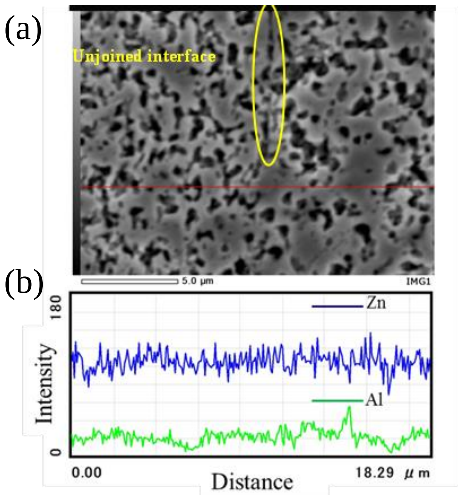

Fig. 5.10 Line analysis carried out on an etched specimen after FSF: (a) micro photo after FSF where interface is thought to have disappeared. The line just below the interface indicates the location of the EDS analysis, (b) the continuous line analysis

A line analysis by energy-dispersive spectroscopy (EDS) was conducted in the interface area where the two sheets of Zn-22Al alloy meet. The EDS line analysis is continuous where the interface is considered to have disappeared. This continuous profile along with the microstructure observations strengthens the possibility of disappearance of the interface. Figure 5.10 shows the EDS results.

5.3.4 Temperature measurements

Increasing the temperature by lowering the revolutionary pitch results in a better plastic flow, but raising the temperature too high can melt the polyimide that coats the wire. The temperature also affects the microstructures of the processed zone and, more importantly, the proper temperature results in joining of the Zn-22Al sheets. For these reasons, the temperature during the process is one of the important parameters in developing the composite. The temperature was measured in different points, as shown in Fig. 5.11.

Fig. 5.12 shows the temperature distribution after the FSF for different travel speeds. When the rotation speed is set to 440 rpm ("0" is the centre of the tool). The temperature clearly increases with decreasing travel speed of the tool. The maximum temperature at point C (the temperature above the copper wire where the interface is considered to have disappeared) was 262°C for travel speed of 50 mm/min. This temperature is close to the temperature having the highest *m*-value (strain rate sensitivity index) and superplasticity for Zn-22Al alloy [167]. This result suggests that SPF/DB occurs in this area. As indicated earlier in previous chapter, a more detailed study of the superplastic mechanism is needed to prove that suggestion. Temperatures at points A and E decrease toward the advancing side (AS) and the retreating side (RS). These points are far from the stirring and forming area, and the lack of heat and amount of tool force are considered to be the reason for the interface still appearing in these areas.

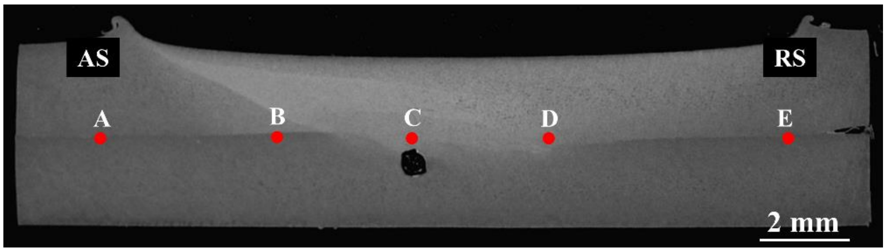

Fig. 5.11 Macro-photo of cross section indicating points (A–E) where temperature was measured during the FSF process with rotation speed of 440 rpm, travel speed of 15–400 mm/min, and counterclockwise tool rotation. Depth of the measured points is 2 mm from the surface on the interface between the upper and bottom sheets of Zn-22Al

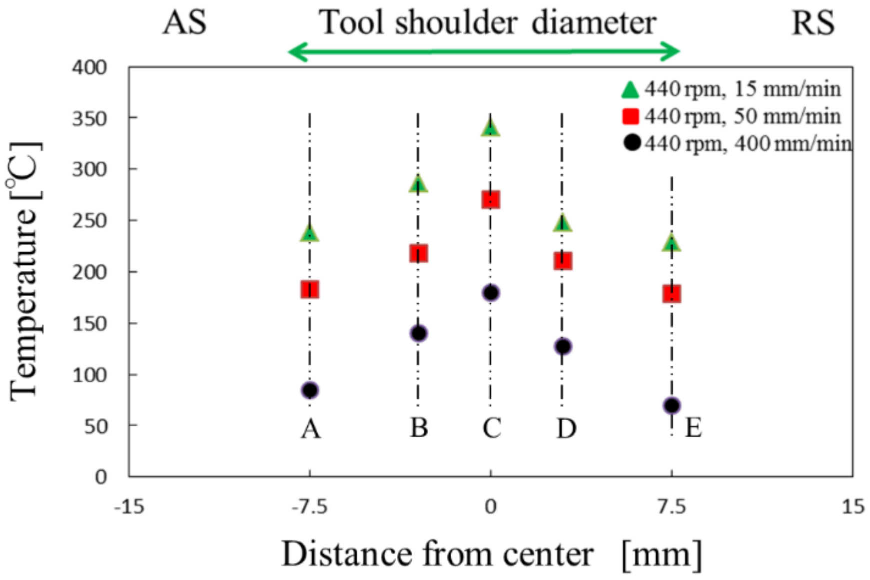

Fig. 5.12 Temperature distribution after FSF for different travel speeds of the FSF tool

5.3.5 Measurement of insulation resistance

A circuit tester was used to check whether the polyimide coating of the wire was broken or short-circuited and whether any insulation was broken. An analog insulation resistance tester using DC 1000 V of electricity was applied to the copper wire to measure the amount of insulation voltage.

The insulation resistance tester can measure up to 1000 V. It was judged that the insulation resistance inside the metal is infinite if the insulation voltage becomes 1000 V. Therefore, it is thought that the transmission of energy or electric signals is possible. The results for different process parameters are shown in Table 5.3.

Polyimide tube (inner diameter: 0.5mm, thickness: 0.06mm) with a heat-resistance temperature of 400°C was used in the experiments. The temperature varies depending on the process parameters and rotation pitches. The heat input increases for smaller rotation pitches. Therefore, at certain process parameters, polyimide is more likely to be destroyed. Polyimide fracture is also seen in the very low heat-input parameter when the travel speed of the tool is too high (for a rotation speed of 315 rpm). The material flow is poor when the temperature is too low, and both the material and the polyimide would be considered to have heterogeneous deformation. Therefore, polyimide would become thinner in some parts and fracture. As a result, no insulation would exist between the copper and the alloy.

Table 5.3 Effect of process parameters on insulation resistance
(Successful parameters: ○, unsuccessful parameters: ×)

Rotation speed / Travel speed	315 [rpm]	440 [rpm]	620 [rpm]	880 [rpm]
25 [mm/min]	○	○	×	×
50 [mm/min]	○	○	○	○
100 [mm/min]	○	○	○	○
200 [mm/min]	○	○	○	○
400 [mm/min]	×	○	○	○

5.3.6 Hardness and grain size measurements

To find and evaluate the effect of the FSF process on the material and its microstructures, hardness tests and grain size measurements were conducted. Fig. 5.13 shows the hardness distribution for different distances from the surface of the FSF cross section. The measurements were taken at 1.5 mm intervals extending horizontally. A difference can be seen in the vertical direction: the area close to the surface tends to be harder. Also, areas under the probe are harder than areas under the shoulder. It is confirmed that FSF has a high effect on hardening the material through the stir zone. The stir zone (maximum hardness about 132 HV) is considerably harder than the base metal (average hardness 107 HV). This can be attributed to the grain refinement due to stirring. Fig. 5.14 shows the average grain size distribution at 0.5 mm under the surface of the cross section. It is confirmed that the stir zone has a considerably finer grain size compared to that of the base metal. The average grain size of 0.7 µm for the base metal was reduced to 0.4 µm within the stir zone. As shown in Fig. 5.13 and Fig. 5.14, the grain size is related to hardness: that is, the hardness increases as the grain size decreases.

In general metallic materials, yield stress (σ_y) is related to grain size (d) through the Hall-Petch equation [149, 150]:

$$\sigma_0 = \sigma_y + k_y d^{-1/2} \qquad (5.1)$$

where σ_0 is the friction stress and k_y is a positive constant of yielding associated with the stress required to extend dislocation activity into adjacent unyielded grains. This relation demonstrates that yield stress increases with decreasing grain size. In the absence of appreciable work hardening, the hardness HV of the material is proportional to the yield stress through the expression HV=3σ_y. Eq. (1) is therefore reformulated in terms of hardness by the following relation [152]:

$$HV = H_0 + k_H d^{-1/2} \qquad (5.2)$$

where H_0 and k_H are the appropriate constants associated with the hardness measurements.

In the present study, hardness increases toward the stir zone and supports the fact that microstructures within the stir zone are finer than those in the base metal. The relation between grain size and hardness in different parts of the processed area (base metal toward stir zone) is shown in Fig. 5.15. The hardness is directly proportional to the grain size, and the Hall-Petch relation of hardness could be found in the friction stir formed Zn-22Al. The relation calculated by the least squares method is expressed in the following equation:

$$HV = 40 + 58\, d^{-1/2} \qquad (5.3)$$

where d is the grain size in micrometres.

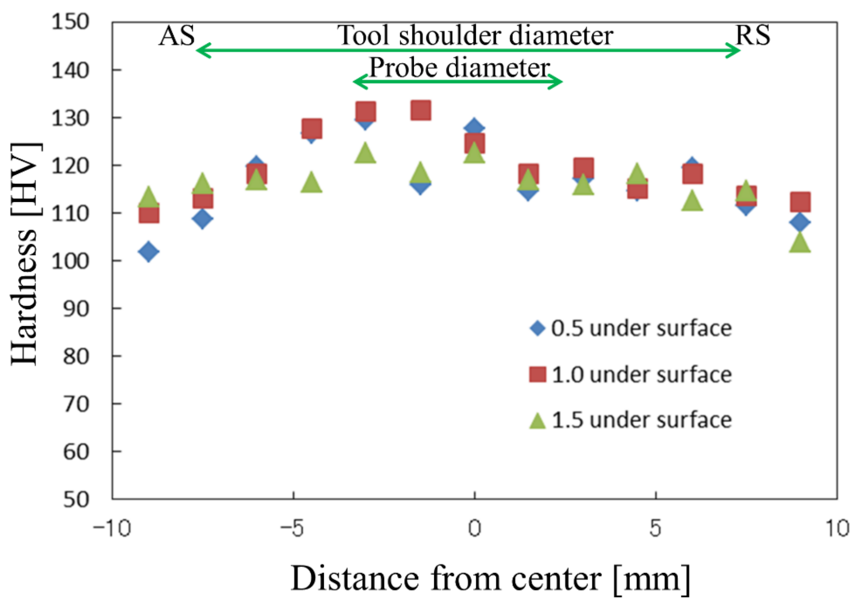

Fig. 5.13 Hardness distribution of cross section after FSF showing that that the hardness increases in the stir area after FSF

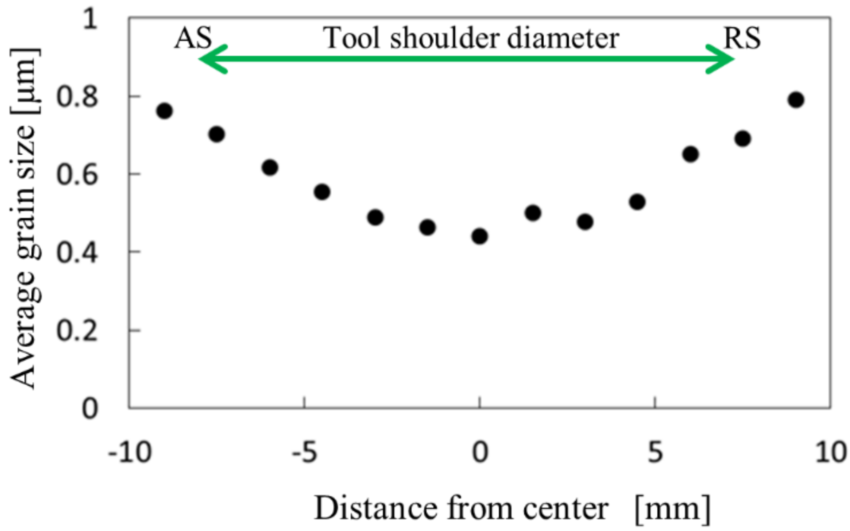

Fig. 5.14 Average grain size distribution in FSF cross section

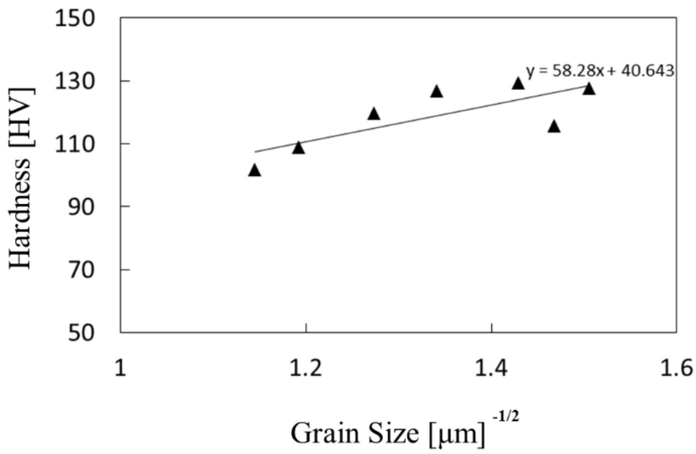

Fig. 5.15 Hall-Petch relation for friction stir formed Zn-22Al as a function of $d^{-1/2}$

The hardness within and around the copper wire was measured as well. Locations of the measurements are shown in Fig. 5.16 and the results are shown in Table 5.4. It is confirmed that the hardness above the wire, which is considered to be the TMAZ part of the FSF process, is harder than the bottom part. The average hardness of the wire is 91 HV after FSF, as compared with 103 HV before the process. Since copper has a somewhat low recrystallization temperature (140 °C) [191], it is concluded that the effects of heat annealed the copper wire from its previous state.

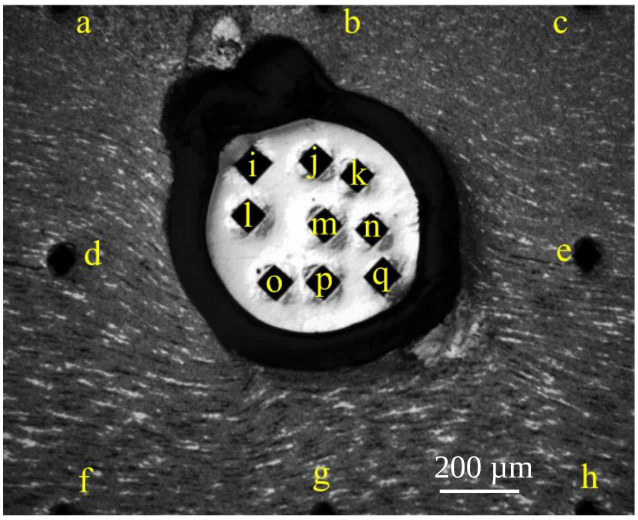

Fig. 5.16 Measurement positions of hardness distribution in the copper wire

Table 5.4 Hardness distribution in insulated copper wire after FSF

Position	Hardness [HV]	Position	Hardness [HV]
a	114	i	99
b	101	j	94
c	114	k	95
d	108	l	82
e	94	m	86
f	102	n	90
g	99	o	95
h	95	p	84
		q	98

5.4 Conclusions

The results of mechanical interlocking of insulated copper wire with Zn-22Al superplastic alloy by the FSF technique were investigated. The following results were obtained.

1. It was confirmed that FSF can be used to form mechanical interlocks between Zn-22Al superplastic alloy and thin insulated copper wire.
2. With proper process parameters of the FSF tool, the temperature of the process can be controlled so that the Zn-22Al alloy can reach its highest superplastic condition. As the result, two sheets of Zn-22Al can be joined to each other. This shows the great possibility of superplastic forming and diffusion bonding between two sheets.
3. It was concluded that FSF can improve the mechanical properties of superplastic alloys due to grain refinement and hardening effects within the processed zone**.** The maximum hardness of the stir zone was 132 HV after FSF, as compared with 107 HV before treatment.
4. The relation between hardness and grain size was expressed by the Hall-Petch relation.
5. FSF offers a high possibility of developing new composite materials for transmitting electricity, electric signals or energy.

Chapter 6

FRICTION STIR FORMING FOR MECHANICAL INTERLOCKING OF Zn-22Al SUPERPLASTIC ALLOY WITH STAINLESS STEEL STRANDS

6. FRICTION STIR FORMING FOR MECHANICAL INTERLOCKING OF Zn-22Al SUPERPLASTIC ALLOY WITH STAINLESS STEEL STRANDS

6.1 Introduction

In this chapter, a novel method of mechanical interlocking of ultra-thin stainless steel strands with Zn-22Al superplastic alloy which was conducted by using friction stir forming (FSF) is introduced.

Fiber reinforced materials (FRM) has gathered intention because of their outstanding properties like specific strength, heat resistance and wear resistance. However, regardless of the mentioned excellent properties, there are a few problems like occurring brittle compound at the interface between the fiber and the metal matrix which makes it difficult to produce FRMs. Moreover, fabrication of aluminum alloys and superplastic matrix composite has recently gathered interests. Nishimura et al. published a study on manufacturing Zn-22Al superplastic matrix composite using powder metallurgy. [97] They developed a compacting method for fabricating metal matrix composite using bondable Zn-22Al alloy powders and SiC fibers. They applied heat treatment for controlling properties of the composite. In their method, requirements of controlling of fabricating temperature and amount of applied pressure, also duration time were needed, which make their method not efficient enough as it is too time consuming, complicated and not so ECO friendly.

Considering mentioned challenges in this field, we applied FSF as a novel method to mechanically interlock stainless steel strands with Zn-22Al alloy and produce a superplastic matrix composite. The trade name of the stainless steel fibers, manufactured by “Nakayama companies” with their original technology is “NASLON”. Stainless steel fiber, as its name would suggest, uses stainless steel as its base, and is a type of metallic fiber. Accordingly, it has completely different properties from conventional organic fibers. Stainless steel fiber's greatest characteristic is that it possesses the same level of workability as fiber while having metallic properties. It can be used for electrostatic applications such as sewing thread for sewing, reinforcement material of FRP · FRM, packing material, heating element, static elimination brush etc.

In present study the potential for development of a multi-functional composite material is studied experimentally. By using FSF technique, it can be expected that stainless steel strands can mechanically get joined to Zn-22Al alloys, and be capable of improving the mechanical properties of the alloy. Moreover, stainless steel strands can be expected to affect the flexibility of the produced composite. The results are discussed in terms of microstructure observations, hardness distributions and tensile tests.

6.2 Experimental Procedure

The FSF system and FSF tool are same as in previous chapters. Experiments are performed on 2 mm thick Zn-22Al as-received alloy. Fig. 6.1 (a) illustrates mechanical interlocking of the stainless steel strands using FSF. As it is shown in Fig. 6.1 (b), a guide slit is cut into the plate by machining and strands (diameter 12μm×100) are put into the guide slit (mechanical properties of the stainless strand is given at table 6.1). Depth of the guide slit is 1.7mm and the width of it is 0.5mm. Then, the FSF is applied to the sheet. To perform FSF, a rotating tool with a profiled probe, is plunged and fed along the line of the slit (Tool rotation is counterclockwise and the plunge depth is set to 1.4mm). The rotating tool is traversed at a 3°forward angle through the slit line. As the tool moves, friction stirring occurs and the work-piece flows into the guide slit as a result of plastic flow. This leads to mechanically interlocking the strands in the middle. Process parameters of FSF tool are shown in table 6.2.

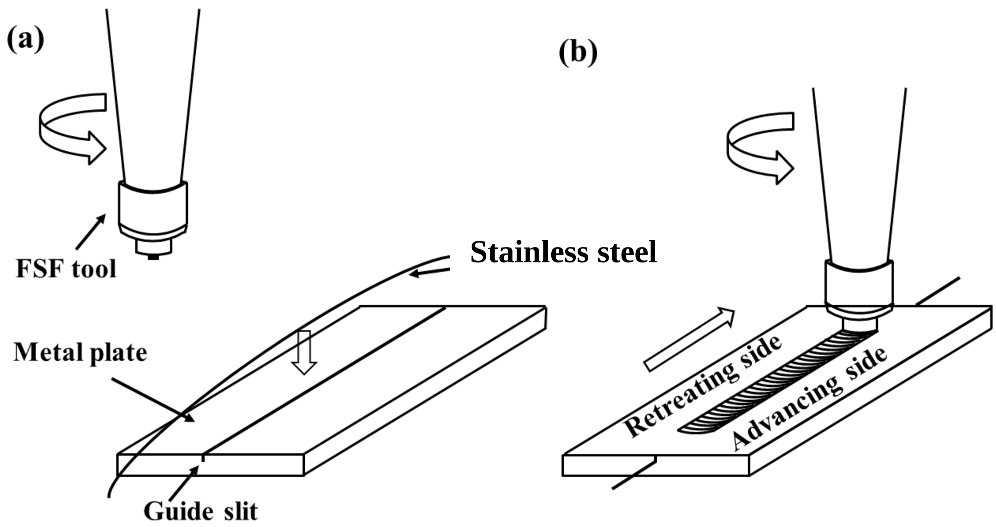

Fig. 6.1 (a) stainless steel strands (12μm×100) are placed into a slit on an Al alloy, (b) Rotating tool is plunged and the moves toward the slit line to perform FSF

Table 6.1 Mechanical properties of the stainless steel strand [192]

Material	Basis weight [g/m]	Tensile strength [N]	Elongation [%]
SUS304	0.11	18	1.0

Table 6.2 Process parameters of FSF tool during the process

Rotation speed [rpm]	Travel speed [mm/min]	Plunge depth [mm]	Tilt angle [°]
880	100	1.4	3

6.3 Results and discussions

6.3.1 Interlocking of Stainless steel strands and Zn-22Al alloy

Fig. 6.2 shows cross sectional photos showing mechanical interlock formed between strands and Zn-22Al. Cross section is perpendicular to the direction of processing and is etched with sodium hydroxide. It can be confirmed that the material has flowed and completely filled the slit. It can be confirmed from Fig. 6.3 that Zn-22Al has flowed and precisely filled the slit and spaces between strands. It should be noted that tool probe passes exactly above the strands therefore, strands are embedded exactly under the stir zone. Since the temperature during friction stir process is relatively low, it would be unlikely to observe any reaction between strands and Zn-22Al alloy or obtain any intermetallic layers between the alloys Fig. 6.4 shows how FSF process affected

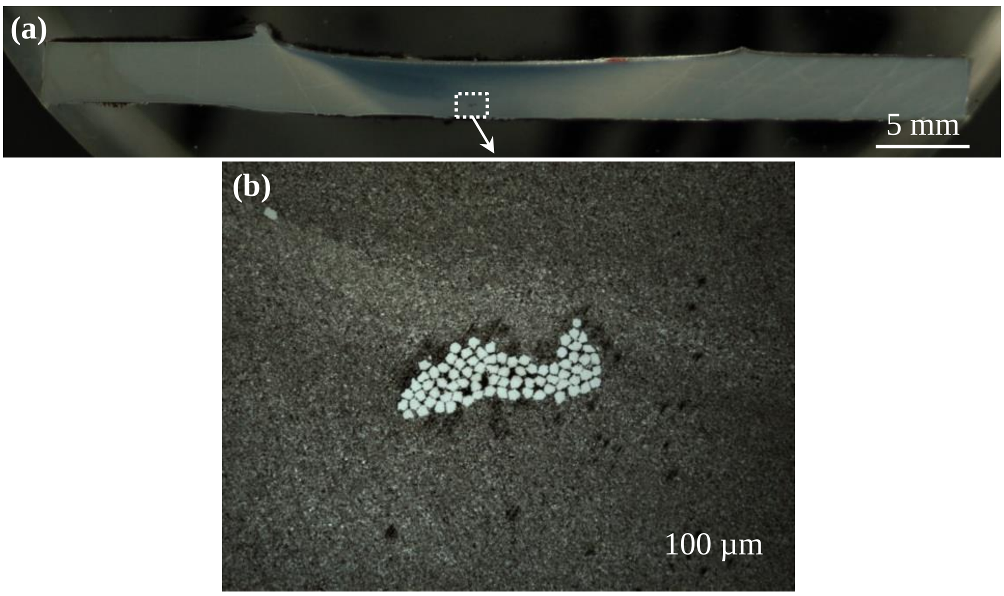

Fig. 6.2 (a) Cross-sectional photo of mechanically interlocked strands formed by FSF for Zn-22Al (b) micro photo of interlocked strands after FSF

the base metal. It can be confirmed that notably finer grain structures has been obtained within the stir zone. Fig. 6.4. (c) shows the TMAZ part of the process.

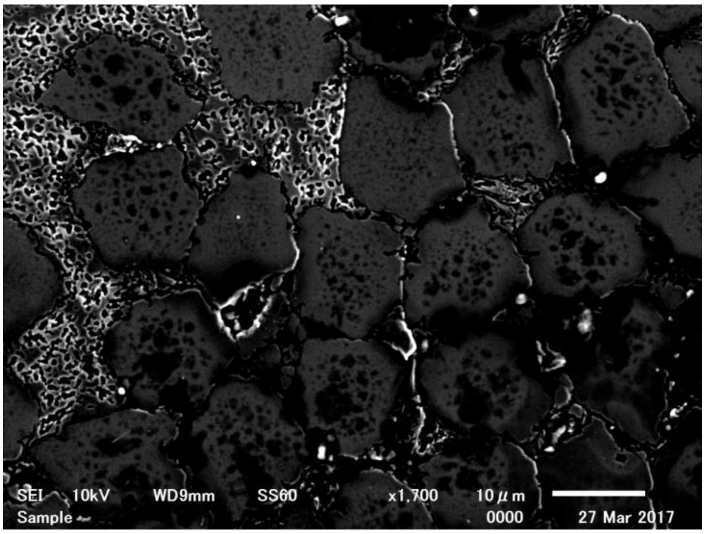

Fig. 6.3 SEM image of interlocked strands showing that material has precisely filled the slit and space between strands

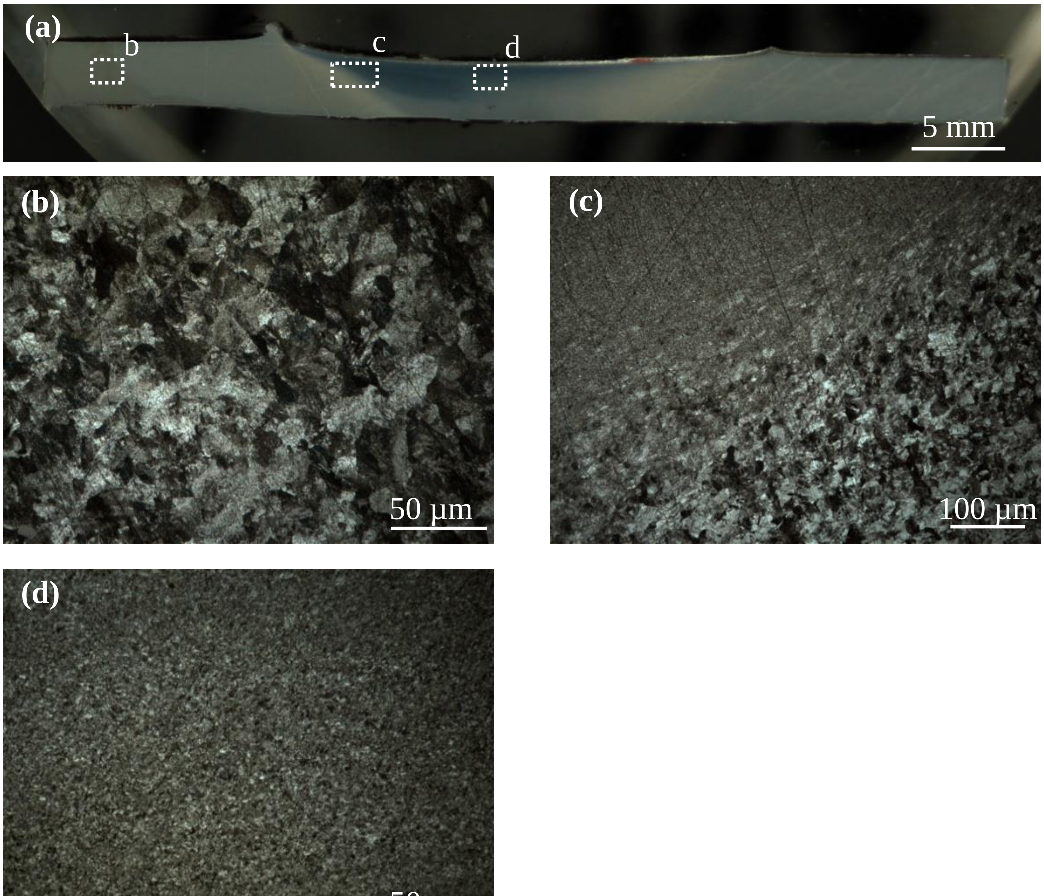

Fig. 6.4 (a) macro photo of cross section after FSF process with microstructures after FSF process showing (b) unaffected zone (base metal) (c) TMAZ (thermo-mechanically affected zone) (d) stir zone

6.3.2 Pull out test

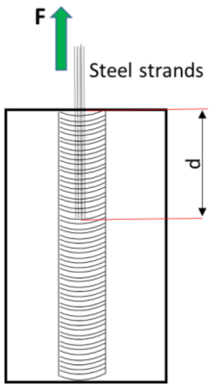

Fig. 6.5 Schematic of experimental setup for pull out

Strength of the interlock was investigated by pulling out tests. K. Yamamura previously showed that the strength of the interlock between piano wire and aluminum alloy by using FSF techniques, is strongly dependent on contact surface area [11]. To test this hypothesis in this study, pull-out tests were carried out with various embedded wire lengths (h) using the experimental setup shown in Fig. 6.5. Strands broke at all embedded lengths. A representative test result (load-

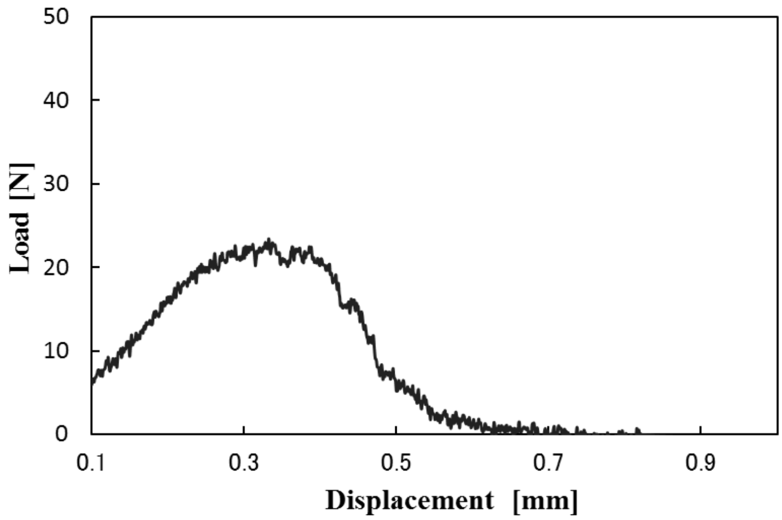

Fig. 6.6 Representative load-displacement graph for friction stir formed Zn-22Al alloy

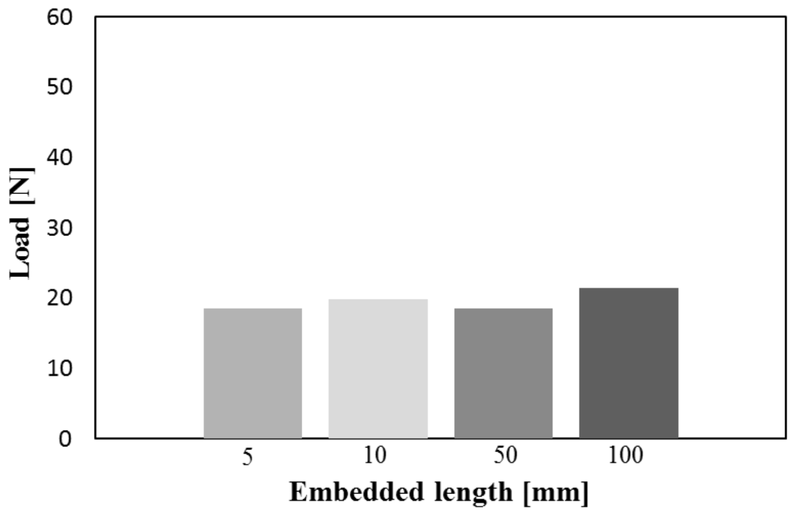

Fig. 6.7 Effect of embedded length on maximum load in pull-out test

displacement curve) is shown in Fig. 6.6. From Fig. 6.7 it can be recognize that there is no noticeable changes in maximum load for different embedded lengths. It can be confirmed that the maximum load of strands after the process close to the strength of strands before applying FSF.

6.3.3 Hardness test

Fig. 6.8 shows the hardness distribution for different distances under the surface of the FSF cross-section. The measurements have been taken at 2.0 mm intervals extending horizontally. It can be confirmed that FSF has a high effect on hardening the material through the stir zone. Stir zone is considerably harder than the base metal. As discussed earlier, this can be attributed to the grain refinement of Zn-22Al due to stirring. However, for some other aluminum alloys softening is apparent in the areas under the shoulder; this softening is due to heat-induced over-aging [11] and it been reported that this condition can be improved through aging treatment [193]. Fig. 6.8 (b) shows the hardness distribution within the stir zone. It can be confirmed that hardness increases toward the surface of the processed area.

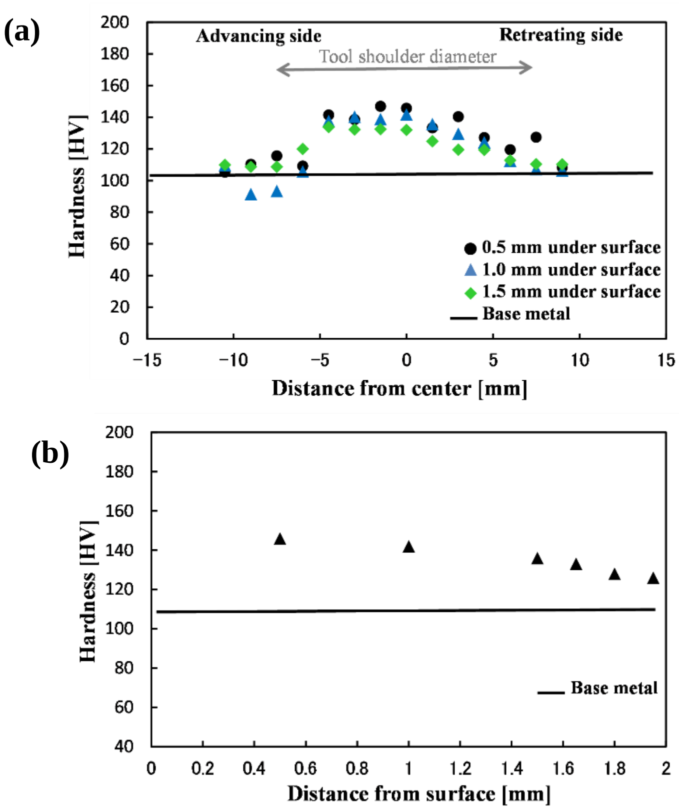

Fig. 6.8 Hardness distribution of cross-section after FSF
(a) different distances from center extending horizontally
(b) different distances from the surface within the stir zone

6.3.4 Tensile tests

Number of slits and strands were increased to investigate the effect of strands number on the mechanical properties of the material. Tensile tests were carried out to evaluate the results. Different number of strands (1 to 5 times of the original 12μm×100) were put into one slit and FSF was applied on top of the slit. Fig. 6.9 shows the macro photo after cross-section. The relation between number of strands and maximum stress is shown in Fig. 6.10. An increasing tendency of strength could be confirmed until the number of strands were increased to two times of the original number, and then strength started to decrease as the number of strands reached five times of the original number. Increasing strands number in an excess amount, causes a not sufficient material flow; Empty spaces between strands and material could remain as a result, therefore, affecting strength of material when subjected to tensile tests.

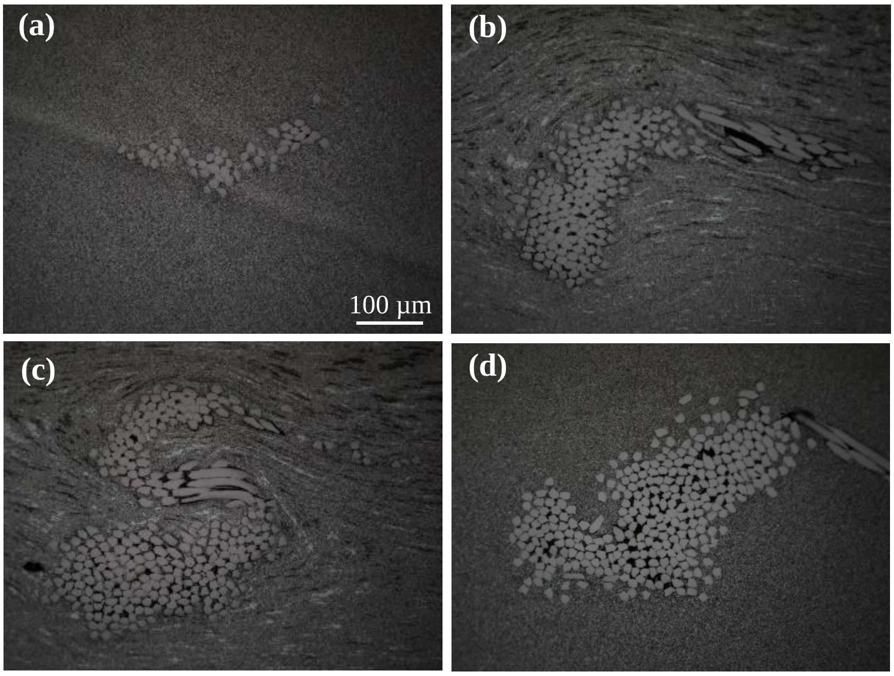

Fig. 6.9 Increasing the number of embedded strands in one slit: (a) 12 µm×100, (b) 2 times of (a), (c) 3 times of (a), (d) 5 times of (a)

Slit numbers in which the strands are embedded are changed from 0 to 5, and the influence was examined by a tensile test after 1 pass of FSF. Relation between the number of embedded strands in multiple slits and the tensile strength is shown in Fig. 6.11. Comparing to the base metal tensile strength increases after applying FSF. This can be linked to the grain refinement after applying friction stir process. As the number of slits increases, the tensile strength tends to increase as well. However, a sudden drop in strength was observe in 5 slit specimens. This could be attributed to lack of material flow into the side slits after applying only 1 pass of FSF (same phenomenon as happened after increasing strands number which was indicated above). Material could not completely flow into the slits on the advancing side (AS) and retreating side (RS) side. As can be seen in Fig. 6.12 (a), the material flow is insufficient due to the heat deficiency when getting away from the center of the tool, therefore material cannot fully flow into the slit. In other hand it can be confirmed from Fig. 6.12 (b) that material can precisely fill the slit when strands are placed exactly under the probe part of the tool.

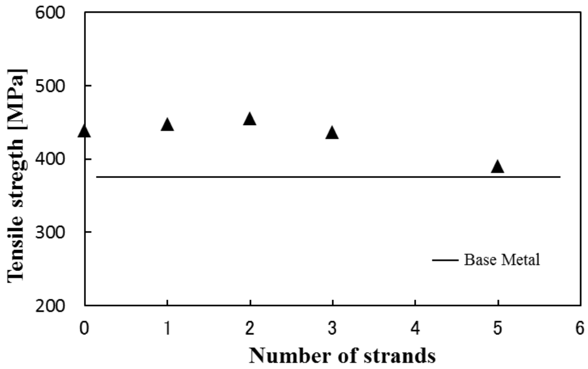

Fig. 6.10 Effect of strands number on maximum stress

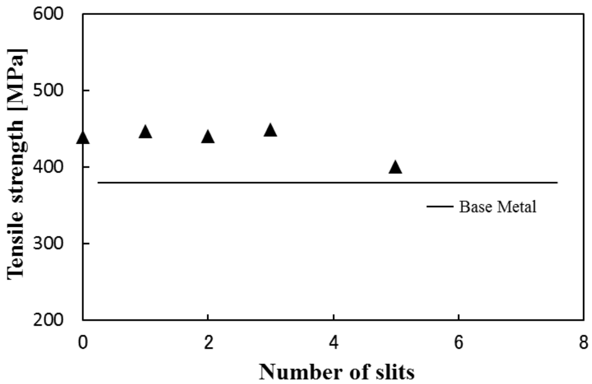

Fig. 6.11 Effect of slit numbers on maximum stress

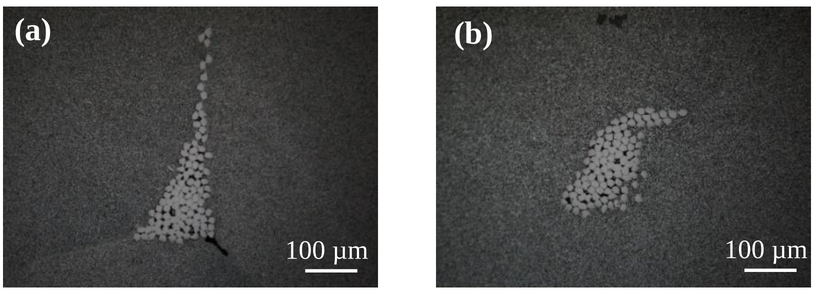

Fig 6.12 (a) lack of material flow in slit 5.0 mm away from the center of tool (b) slit under the center of tool has been filled completely

According to the microstructural observation using SEM, although a difference in how the plasticized material behave and flow in AS and RS was observed; when the number of slits increased to 5, a lack of material flow was confirmed in both AS and RS (in slits 5 mm away from the center) and slits were not fully filled in both sides. As it is shown in Fig. 6.13, SEM images were taken after tensile test for base metal, friction stir processed alloy without any strands and friction stir formed alloy with interlocked strands. Dimples can be observed for all the taken samples. It can be confirmed from Fig. 6.13 (c) that crack occurs in the slit which strands were embedded and as it can be confirmed in Fig. 6.14, some strands tended to remain unharmed and not break after the tensile test. This can bring a possibility of using strands to produce a flexible joint assembly.

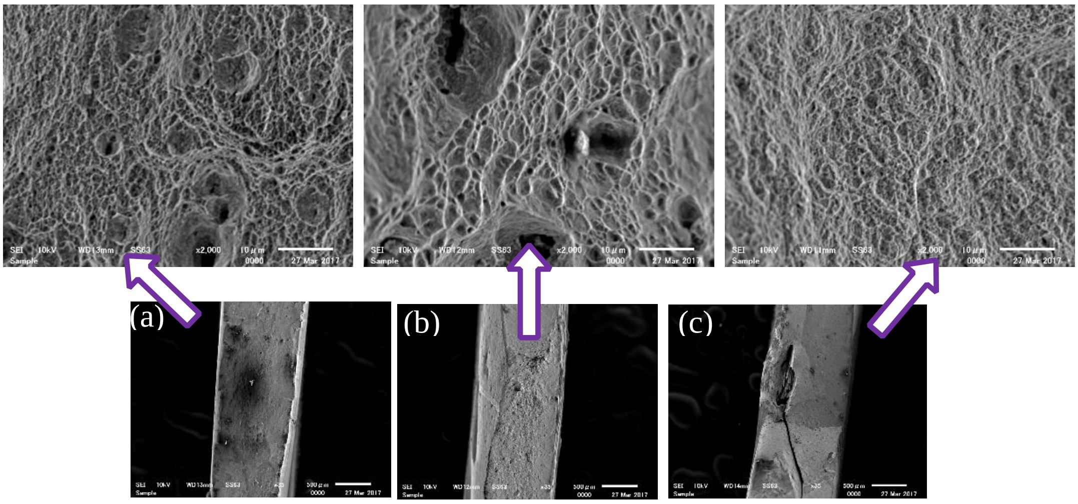

Fig 6.13 SEM images after tensile test showing (a) base metal (b) friction stir processed alloy (c) friction stir formed alloy with strands

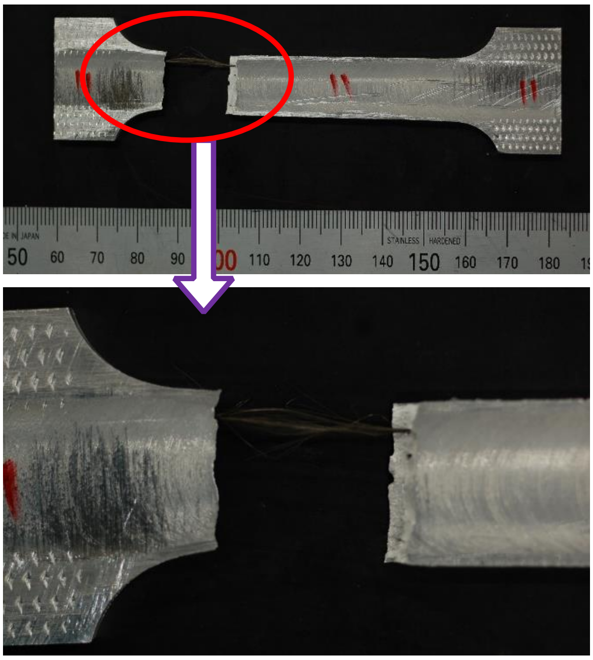

Fig 6.14 Non-broken strands still remaining after tensile test

6.3.5 Developing an interlocked bulk superplastic composite

FSF was carried out in several passes by offsetting tool, to develop a superplastic composite plate interlocked by stainless steel strands. As shown in Fig. 6.16, slits are cut into a Zn-22Al plate by machining and strands (diameter 12μm×100) are put into the slits in the intervals of 5mm. Depth of the guide slit is 0.8 mm and the width of it is 0.5mm. Then, another Zn-22Al plate is place on top and the FSF is applied to the upper sheet. strands were placed in slits 0° and 45° angles to the direction of the processing and multi-passes of FSF

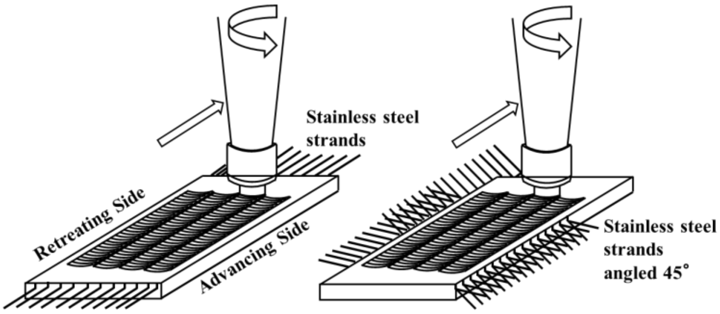

Fig. 6.16 Schematic illustration showing stainless steel strands position in alloy

were carried out.

Fig. 6.17 shows macro photos of cross section after friction stir lap welding of Zn-22Al alloys without any interlocked strands (a) and with interlocked strands (b). Hara had previously succeeded in producing a bulk superplastic material [194]. He reported about the improvement of mechanical properties after friction stir lap welding of Zn-22Al and how process parameters affect properties of the assembly.

To evaluate the properties of the developed plate, tensile tests were carried out.

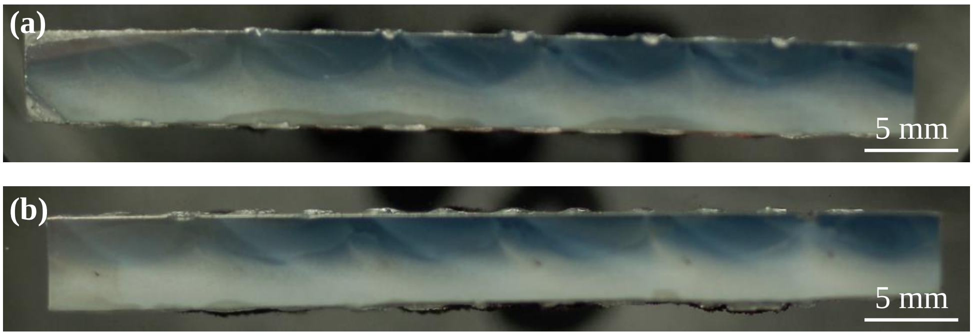

Fig 6.17 (a) Macro photo of cross-section of a friction stir lap welded Zn-22Al as-received alloy (b) macro photo of friction stir lap welded Zn-22Al with interlocked strands

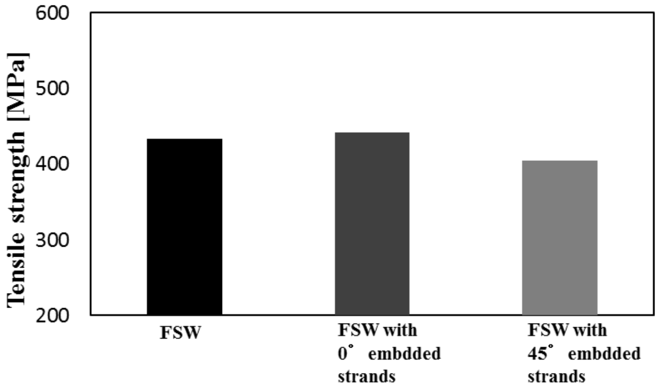

Fig. 6.18 Tensile test results after friction stir lap welding with and without strands

Fig. 6.18 shows how the interlocked strands in different directions affect tensile strength. Embedding strands in a 0° direction resulted in a higher strength while strength decreased for 45° strands. Fig. 6.19 is a SEM image of a tensile sample fracture surface for a friction stir formed Zn-22Al with interlocked strands. Observation of the strands position after tensile tests revealed that in 45° embedded strands, empty spaces remain in slits due to lack of material flow that results in a inhomogeneous grain structure which leads to a dropped strength. Excessive number of slits in a single plate inhibits sufficient material flow to fill the slits, which makes the final produced composite vulnerable to stress. However, as it can be seen in figure 6.20, fine dimples could be confirmed after SEM observation. Future works could challenge to solve the mentioned problems by decreasing the number of slits along with decreasing the tool offset distance, also considering apply of overlapped multi-passes of FSF.

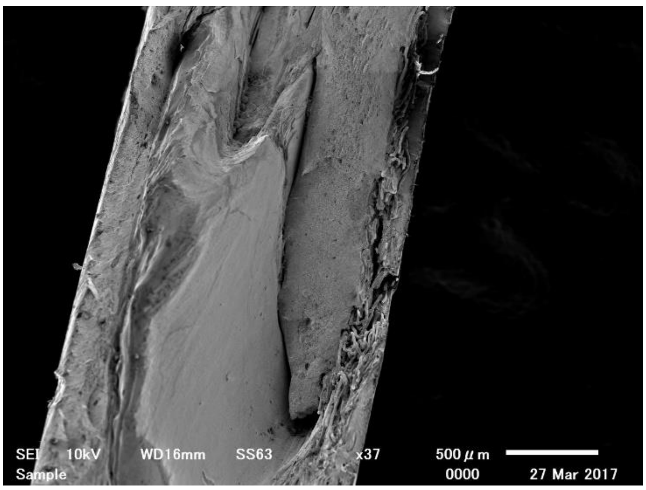

Fig. 6.19 SEM image after tensile test for a 45° embedded strands sample in a friction stir formed Zn-22Al.

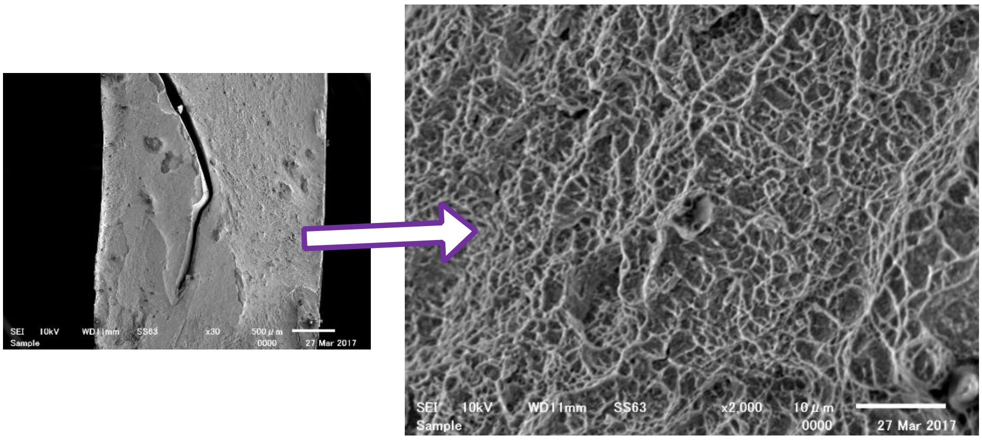

Fig. 6.20 SEM image after tensile test showing fine dimples

Conclusions

It was confirmed that FSF can be used as a novel method to form mechanical interlocks between Zn-22Al superplastic alloy and ultra-thin stainless steel strands and develop a multi-functional composite alloy. Moreover, results of mechanical interlocking of an insulated copper wire with Zn-22Al superplastic alloy were investigated by using the FSF technique and the following results were obtained.

1. Material hardening was observed in the stirring part as a result of grain refinement due to the stirring process during FSF.
2. Strands tended to break when pulled and not get pulled-out when subjected to pull-out tests. It was confirmed that maximum load of the strands in developed assembly is not dependent on the embedded length of the interlocked strands.
3. Tensile strength increases after applying FSF while elongation was decreased. Grain refinement of grain structure due to FSF could be linked to the mentioned result. Higher elongations can be expected in high temperature testing condition.
4. An increasing tendency of strength could be confirmed by increasing the number of strands in one slit. However, putting strands in multiple slits resulted in insufficient material flow. It was suggested that the insufficient flow of material can be improved by increasing the heat-input during the FSF process by changing the process parameter of the tool or applying FSF in multi-passes, which can be raised as future challenges for present experiment.

Chapter 7

CONCLUSIONS

7. CONCLUSIONS

7.1 A brief summary

In the present study, friction stir processing (FSP) was presented as a technique to improve the mechanical properties of Zn-22Al superplastic alloy. The effect of FSP on the superplastic behavior of Zn-22Al was discussed in Chapter 1. FSP as a forming technique, referred to as "friction stir forming" (FSF), was used on Zn-22Al to produce multi-functional superplastic alloys.

In chapter 2, an analytical overview of the literature published on the topic was produced. In chapter 2 we reported our critical review of the relevant literature and addressed gaps in those literatures. We introduced methods that how our research attempts to address those gaps.

In Chapter 3, the effect of FSP on the microstructure of Zn-22Al superplastic alloy was proposed. In particular, the effect of varying the different tool process parameters of FSP on the grain size of superplastic Zn-22Al was discussed. Based on microstructural observations and hardness tests, it was concluded that grain refinement of Zn-22Al alloy by FSP is possible; therefore, mechanical and superplastic properties can be improved.

As discussed in Chapter 3, a grain size of 0.3 µm was achieved within the stir zone. Hardness of Zn-22Al increased toward the stir zone after FSP as a result of grain refinement. Tensile tests after FSP revealed the processed alloy has a greater *m*-value and superplasticity can be obtained at a higher strain rate of 1×10^{-1}.

FSF as a forming technique was used as a novel method to produce multi-functional composite alloys.

In Chapter 4, a superplastic vibration damping composite was developed by using FSF. Temperature measurements during FSF showed that, by changing the process parameters of the FSF tool, the temperature can be controlled. Therefore, by controlling the process parameters and the temperature, Zn-22Al can reach its highest superplastic condition during the FSF process. The possibility of diffusion bonding by superplastic forming during FSF was raised and it was demonstrated that FSF can successfully interlock perforated steel sheet between layers of Zn-22Al and develop a superplastic damping fiber. The damping ability of superplastic vibration-damping steel sheet composite was 1.7 times larger than the damping ability of Zn-22Al, a well-known damping alloy.

In Chapter 5, the same FSF technique was used to produce a multi-functional superplastic composite capable of transmitting data or energy by mechanically interlocking an insulated copper wire to Zn-22Al alloys. The FSF technique was also used to develop a new superplastic composite. We noted the potential scientific impact of the friction stir based technique in future studies by other scientists investigating mechanical and airtight interlocks.

Chapter 6 scientifically demonstrated a novel method for producing MMCs by mechanically interlocking stainless steel strands and Zn-22Al alloy and for developing a multi-functional composite alloy. This chapter described the possibility of using FSF as a novel method for joining dissimilar alloys to develop a new composite. Moreover, stainless steel strands can be expected to affect the flexibility of the produced composite. It was confirmed that the maximum pull-out load of the developed assembly is not dependent on the embedded length of the interlocked strands. Stainless steel strands tend to break under tensile tests instead of getting pulled out. An increasing tendency of strength could be confirmed by increasing the number of strands in one slit. However, putting strands in multiple slits resulted in insufficient material flow.

7.2 Recommendations for future research

The results of this study opened a channel for discussing possible new applications of FSP, such as applying FSP for improvement of mechanical properties of Zn-22Al superplastic alloy and development of fine-grained structures for increasing superplasticity. Moreover, FSP was introduced as a novel method of creating innovative functions for Zn-22Al superplastic alloy. Producing multi-functional superplastic materials was studied experimentally through FSF. These achievements demonstrate that this research is important for researchers and practitioners. By combining FSP and superplastic phenomena of Zn-22Al alloy, new method of producing superplastic composites with improved mechanical and superplastic properties were introduced.

A particular finding in this research was grain refinement of superplastic alloy by using friction stir processing. This finding can be beneficial for different fields of studies. However, a few problems were discovered during experiments. An inhomogeneous microstructure after applying FSP to the alloy was one of the problems in our study. Overlapping passes of FSF to develop a processed plate have an effect on reducing inhomogeneous structures. Other studies have mentioned that applying FSF in multi-passes has an advantage over only a single pass of FSP and can result in a more homogeneous structure. Therefore, applying FSP in multi-passes can be of interest in future research to produce even more fine-grained structures in Zn-22Al alloys.

The finding in our research that was not anticipated was that joining sheets of Zn-22Al alloy was possibly due to SPF/DB. Chapter 4 included discussions on producing superplastic damping fiber by using FSF, and joining two sheets of Zn-22Al to each other as the FSP tool moves along the upper sheet without touching the sheet at the bottom. Temperature measurements were carried out and it was found out that at specific process parameters during FSP, the temperature can get close to the recrystallization temperature of Zn-22Al alloy and therefore SPF/DB occurs and the upper and the lower sheets are joined together. More detailed study is needed to prove this idea. Future attempts are expected to focus on the study of the grain boundaries of Zn-22Al for different process parameters of FSP. This can result in desirable findings that can have a beneficial effect in superplastic forming industries.

Limitations include temperature measurements during friction stirring processes. Although temperature is an important factor during the friction stirring process, only a few studies have reported on temperature measurements of the workpiece and the FSW tool during the friction stirring process. [129] In the present study, the temperatures of specific points in the workpiece were measured. K type thermocouples were inserted directly into different parts of the workpiece to measure the exact temperature when the tool passed that specified point. However, the difficulty of measuring temperature in a more continuous manner still remains. Measuring the temperature of different parts of a workpiece and the rotating tool constantly throughout the process can be a huge benefit to researchers and manufacturers.

Finally, the results of this research provide recommendations for practitioners in the field of material processing. For example, FSF which was originally introduced by Nishihara as a new forming method, was applied to superplastic alloys for the first time for manufacturing functional composites, so our study suggests new strategies to explore such findings in the future.

As was discussed and demonstrated in Chapters 4 to 7, FSF has the possibility of creating a composite alloy capable of transmitting energy, electricity, and also information. FSF also enables us to produce MMCs in a non-labor-intensive and non-time-consuming way and also in a more eco-friendly manner. Thus, by interlocking steel strands with superplastic alloy, materials with capability of greater flexibility can be manufactured.

Although the present study focused on Zn-22Al as a superplastic alloy, the novel research strategies in other studies can be used for different alloys in the future. Titanium and its alloys are great candidates for future studies, because they are highly used in aerospace and biomechanical industries. FSF can be used for interlocking strands, insulated copper wires or optical fibers in titanium alloys to produce multi-functional alloys.

REFERENCES

[1] W.M. Thomas, E.D. Nicholas, J.C. Needham, M.G. Murch, P. Temple-Smith, and C.J. Dawes, Friction Stir Welding, (1991) International Patent Application No. PCT/GB92/02203 and GB Patent Application No. 9125978.8, U.S. Patent No. 5460317.

[2] Tadashi Nishihara, Proceeding of Japanese Society for Technology of Plasticity (2000) 457-485.

[3] W. M. Thomas, G. Verhaeghe J. Martin, D. G. Staines and C. Stanhope: INALCO, "Friction stir welding - process variants - an update", Paper presented at INALCO (2007) Japan.

[4] R.S. Mishra "Friction Stir Processing for Superplasticity", Adv. Mater. Process. 162(2) (2004) 45-47.

[5] Nishihara T., "Friction stir welding of superplastic Zn–22Al Alloy," Proceedings of the Fourteenth International Offshore and Polar Engineering Conference 4 (2004) 78–82.

[6] Barnhurst R.J., "Zinc and zinc alloys," Properties and Selection: Nonferrous Alloys and Special-Purpose Materials, ASM Handbook, ASM International. 2 (1990) 542.

[7] Hamilton C.H., Ghosh, A.K., "Superplastic sheet forming," Metals Handbook 9th Edition, ASM International. 14 (1988) 852.

[8] T. Nishihara, Japanese Unexamined Patent Application Publication, No. 2002-256453.

[9] T. Nishihara, "Development of Friction Stir Forming", Mater. Sci. Forum. 426-432 (2003) 2971-2978.

[10] T. Nishihara, A. Ito, "Measurement of Die Temperature during Friction Stir Forming" Weld. World, 49(3/4) (2005) 22-26.

[11] K. Yamamura, K. Torikai, and T. Nishihara, "Mechanical Interlock of Thin Metallic Wire Using Friction Stir Forming", Friction Stir Welding and Processing VI, ed. by R. Mishra, M. W. Mahoney, Y. Sato, Y. Hovanski, and R. Verma, (TMS, 2011) 281-288.

[12] T. Nishihara and K. Yamamura, "Local Manufacture of MMC Using FSW", Mater. Sci. Forum. 539-543 (2007) 3733-3738.

[13] Koshi Yamamura and Tadashi Nishihara, “Development of Local Reinforcement and Local Metallic Foam Using FSP”, Mater, Sci. Forum. 638-642 (2010) 1267-1272.

[14] Takahiro Ohashi, Hamed Mofidi Tabatabaei, Tadashi Nishihara, Observation of Material Flow in Friction Stir Forming for A5083 Aluminum Alloy Gear-Racks, Material Science Forum. 889 (2016) 113-118.

[15] T. G. Langdon: Metall Trans., “The mechanical properties of superplastic materials”, 13A (1982) 689–701.

[16] Pearson, C. E., "The Viscous Properties of Extruded Eutectic Alloys of Lead-Tin and Bismuth-Tin", J. Inst. Metals. 54 (1934) 111-123.

[17] Atul H. Chokshi, Amiya K. Mukherjee, Terence G. Langdon, “Superplasticity in advanced materials”, Mater. Sci. and Eng. R: Reports. 10, Issue 6 (1993) 237–274.

[18] ASM handbooks Volume 14B, Metalworking: Sheet Forming, Superplastic Sheet Forming, Requirements for Superplasticity.

[19] M. Mabuchi, K. Higashi, "Superplasticity in Metal Matrix Composites", Key Eng. Mater 104-107 (1995) 225-240.

[20] Degarmo, E. Paul; Black, J T.; Kohser, Ronald A., Materials and Processes in Manufacturing (9th ed.), (2003) Wiley, ISBN 0-471-65653-4.

[21] T. Tanaka, and K. Higashi, “Superplasticity at Room Temperature in Zn-22Al Alloy Processed by Equal-Channel-Angular Extrusion”, Maters Transactions. 45, No. 4 (2004) 1261-1265.

[22] H. Somekawa, T. Tanaka, H. Sasaki, K. Kita, A. Inoue and K. Higashi, “Diffusion Bonding in Ultra Fine-grained Al–Fe Alloy indicating High-strain-rate Superplasticity” Acta Meter. 52 Issue 4 (2004) 1051–1059.

[23] M. Kawasaki, N. Balasubramanian, T. G. Langdon, “Flow Mechanism in Ultra Fine-grained Metals with an Emphisis on Superplasticity”, Mater. Sci. Eng. A. 528 (2011) 6624-6629.

[24] K. Nakashima, Z. Horita, M. Nemoto and T. G. Langdon, “Influence of channel angle on the development of ultrafine grains in equal-channel angular pressing”, Acta Mater. 46 issue 5 (1998) 1589–1599.

[25] R. S. Mishra, R. Z. Valiev and A. K. Mukherjee, "The observation of tensile superplasticity in nanocrystalline materials", NanoStructured Mater. 9 (1997) 473–476.

[26] V.M. Segal, V.I. Reznikov, E.E.Drobyshevskiy and V.I. Kopylov, "Plastic working of metals by simple shear", Russian Metal 1 (1981) 99-105.

[27] R.Z. Valliev and T.G. Langdon Rev, "Developments in the Use of ECAP Processing for Grain Refinement", Adv.Mater.Sci. 13 (2006) 15-26.

[28] R.Z. Valiev, N. A. Krasilnikov and N.K. Tsenev, "Plastic deformation of alloys with submicron-grained structure", Mater.Sci. Eng. A. 137 (1991) 35-40.

[29] R.Z. Valiev, N. A. Krasilnikov and R.R. Mulyukov, "Structure and properties of ultrafine-grained materials produced by severe plastic deformation", Mater. Sci. Eng. A168 (1993) 141-148.

[30] Y. Nishida, H. Arima, J.C. Kim and T. Ando, "Superplasticity of SiCw/7075 Composites Processed by Rotary--Die Equal--Channel Angular Pressing", J. Japan. Inst. Metals 64 (2000) 1224-1229.

[31] Y. Nishida, H. Arima, J.C. Kim and T. Ando, "Rotary-die equal-channel angular pressing of an Al – 7 mass% Si – 0.35 mass% Mg alloy", Scripta Mater. 45 (2001) 261-266.

[32] A. Ma, K. Suzuki, N. Saito, Y. Nishida, M. Takagi, I. Shigematsu and H. Iwata, "Impact toughness of an ingot hypereutectic Al–23 mass% Si alloy improved by rotary-die equal-channel angular pressing", Mater. Sci. Eng. A399 (2005) 181-189.

[33] A. Watazu, I. Shigematsu, A. Ma, K. Suzuki, T. Imai and N. Saito, "Commercial Purity Titanium Processed by Rotary-Die Equal Channel Angular Pressing Method", Mater. Trans. 46 (2005) 2098-2101.

[34] A. Ma, Y. Nishida, K. Suzuki, I. Shigematsu and N. Saito, "Characteristics of plastic deformation by rotary-die equal-channel angular pressing", Scripta Mater. 52 (2005) 433-437.

[35] A. Azushima and K. Aoki, "Properties of ultrafine-grained steel by repeated shear deformation of side extrusion process", Mater. Sci. Eng. A 337 (2002) 45-49.

[36] K. Nakashima, Z. Horita, M. Nemoto and T.G. Langdon, "Development of a multi-pass facility for equal-channel angular pressing to high total strains", Mater. Sci. Eng. A281 (2000) 82-87.

[37] S. W. Chung, H. Somekawa, T. Kinoshita, W. J. Kim and K. Higashi, "The non-uniform behavior during ECAE process by 3-D FVM simulation", Scr. Mater. 50 (2004) 1079–1083.

[38] P. B. Berbon, S. Komura, A, Utsunomiya, Z. Horita, M. Furukawa, M. Nemoto and T. G. Langdon, "An Evaluation of Superplasticity in Aluminum-Scandium Alloys Processed by Equal-Channel Angular Pressing", Mater. Trans. 40 (1999) 772–778.

[39] H. Watanabe, T. Mukai, K. Ishikawa and K. Higashi, "Low temperature superplasticity of a fine-grained ZK60 magnesium alloy processed by equal-channel-angular extrusion", Scr. Mater. 46 (2002) 851–856.

[40] H. Watanabe, T. Mukai, M. Mubuchi and K. Higashi, "High-strain-rate superplasticity at low temperature in a ZK61 magnesium alloy produced by powder metallurgy", Scr. Mater. 41 (1999) 209–213.

[41] M. Kamachi, M. Furukawa, Z. Horita and T.G. Langdon, "Equal-channel angular pressing using plate samples", Mater. Sci. Eng. A 361 (2003) 258-266.

[42] S. Ferrasse, V.M Segal, S.R. Kalidini and F. Alford, "Texture evolution during equal channel angular extrusion: Part I. Effect of route, number of passes and initial texture", Mater Sci Eng A 368 (2004) 28-40.

[43] S. Ferrasse, V.M. Segal and F. Alford, "Effect of additional processing on texture evolution of Al0.5Cu alloy processed by equal channel angular extrusion (ECAE)", Mater. Sci. Eng. A 372 (2004) 44-55.

[44] S. Ferrase, V.M. Segal and F. Alford, "Texture evolution during equal channel angular extrusion (ECAE): Part II. An effect of post-deformation annealing", Mater. Sci. Eng. A 372 (2004) 235-244.

[45] M. Kamachi, M. Furukawa, Z. Horita and T.G. Langdon, "Achieving Superplasticity of Al-1%Mg-0.2%Sc Alloy in Plate Samples Processed by Equal-channel Angular Pressing", Mater. Trans. 45 (2004) 2521-2524.

[46] Z.Y. Ma, A.L. Pilchak, M.C. Juhas, J.C. Williams, “Microstructural refinement and property enhancement of cast light alloys via friction stir processing”, Scr. Mater. 58 (2008) 361-366.

[47] M.L. Santella, T. Engstrom, D. Storjohann, T.Y. Pan, “Effects of friction stir processing on mechanical properties of the cast aluminum alloys A319 and A356”, Scr. Mater. 53 (2005) 201- 206.

[48] Yong-Jai Kwon, Ichinori Shigematsu and Naobumi Saito, “Production of Ultra-Fine Grained Aluminum Alloy using Friction Stir Process”, Materials Transactions, Vol. 44, No. 7 (2003) 1343-1350

[49] G. Venkateswarlu, D. Devaraju, M.J. Davidson, B. Kotiveerachari, G.R.N. Tagore, “Effect of overlapping ratio on mechanical properties and formability of friction stir processed Mg AZ31B alloy”, Materials and Design 45 (2013) 480-486.

[50] Z.Y. Ma, A.L. Pilchak, M.C. Juhas, J.C. Williams, “Microstructural refinement and property enhancement of cast light alloys via friction stir processing”, Scr. Mater (2008) 58, 361-366.

[51] B.M. Darras, M.K. Khraisheh, F.K. Abu-Farha, M.A. Omar, “Friction stir processing of commercial AZ31 magnesium alloy”, J. of Mater. Proc. Technol. 191 (2007) 77-81.

[52] F. Chai, D. Zhang, Y. Li, W. Zhang, “High strain rate superplasticity of a fine-grained AZ91 magnesium alloy prepared by submerged friction stir processing”, Mater. Sci. & Eng. A 568 (2013) 40-48.

[53] B. Darras, E. Kishta, Submerged, “Friction stir processing of AZ31 magnesium alloy”, Mater. Design 47 (2013) 133-137.

[54] J. Iwaszko, K. Kudla, K. Fila, M. Strzelecka, “The Effect of Friction Stir Processing (FSP) on the Microstructure and Properties of AM60 Magnesium Alloy”, Arch. Metall. Mater., Vol. 61 No 3 (2016) 1555-1560.

[55] Y.C. Chen, K. Nakata, “Evaluation of microstructure and mechanical properties in friction stir processed SKD61 tool steel”, Materials Characterization 50 (2009) 1471-1475.

[56] H. Nami, H. Adgi, M. Sharifitabar, H. Shamabadi, “Microstructure and mechanical properties of friction stir welded Al/Mg2Si metal matrix cast composite”, Mater. Design 32 (2011) 976-983.

[57] Yong-Jai Kwon, Ichinori Shigematsu and Naobumi Saito, “Production of Ultra-Fine Grained Aluminum Alloy using Friction Stir Process”, Mater. Trans. 44, No. 7 (2003) 1343-1350.

[58] Yong-Jai Kwon, Ichinori Shigematsu and Naobumi Saito, “Mechanical Property Improvements in Aluminum Alloy through Grain Refinement using Friction Stir Process”, Mater. Trans. 45, No. 7 (2004) 2304-2311.

[59] J.T. Hayashi, S.K. Menon, J. Q. Su and T.R. McNelley, “Friction Stir Processing (FSP) of As-Cast AA5083 for Grain Refinement and Superplasticity”, Key Eng. Mater. 443 (2010) 135-140.

[60] Haider T. Naeem, Kahtan S. Mohammad, and Khairel R. Ahmad, “Effect of Friction Stir Processing on the Microstructure and Hardness of an Aluminum-Zinc-Magnesium-Copper Alloy with Nickel Additives”, Phys. Met. Metallor. 116, No. 10 (2015) 1035-1046.

[61] Ehab A. Al-Danaf, Magdy M. El-Rayes, Mahmoud S. Soliman, “Friction Stir Processing: An effective technique to refine grain structure and enhance ductility”, Mater. Design 31 (2010) 1231-1236.

[62] Chengqi Wang, ming Sun, Feiyan Zheng, Liming Peng, Wenjiang Ding, “Improvement in grain refinement efficiency of Mg-Zr master alloy for magnesium alloy by friction stir processing”, Journal of Magnesium and alloys 2 (2014) 239-244.

[63] I. Charit and R.S Mishra, “Evaluation of microstructure and superplasticity in friction stir processed 5083 Al alloy”, J. Mater. Res. 19 No 11 (2004) 3329-3340.

[64] N. Saito, I. Shigematsu, T. Komaya, T. Tamaki, G. Yamauchi, M. Nakamura, “Grain refinement of 1050 aluminum alloy by friction stir processing”, J. Mater. Sci. Letters. 20 (2001) 1913-1915.

[65] T.R. McNelly, “Friction Stir Processing (FSP): refining microstructures and improving properties”, Revista De Metalurgia 46 (2010) 149-156.

[66] K. Makii, Y. Seki, A. Kushibe and K. Higashi, “Realizing High Strain Rate Super Plasticity at Room Temperature using Thermo Mechanical Controlling Process”, The Fourth Pacific Rim International Conference on Advanced Materials and Processing (PRICM4) (2001) 1977–1978.

[67] T. Tanaka, K. Makii, A. Kushibe and K. Higashi, "Room Temperature Deformation Behavior of Zn-22 mass%Al Alloy with Nanocrystalline Structure", Mater. Trans. 43 (2002) 2449–2454.

[68] T. Tanaka, K. Makii, A. Kushibe and K. Higashi, "Deformation Behavior of Nanocrystalline Zn-22wt%Al Alloy Using Thermomechanical Controlling Process" Key Eng. Mater. 233–236 (2003) 719–724.

[69] H. Ueda, T. Okada, M. Kato, Y. Mimura, K. Makii, M. Tabuchi, K. Aoki, T. Okamoto, H. Okumoto and A. Kushibe: The second Japan National Symposium on Structural Control., (2000) 545–551.

[70] T. Tanaka, K. Makii, A. Kushibe, M. Kohzu and K. Higashi, "Capability of superplastic forming in the seismic device using Zn–22Al eutectoid alloy", Scr. Mater. 49 (2003) 361–366.

[71] R.J. Barnhurst, Zinc and zinc alloys, Properties and Selection: Nonferrous Alloys and Special-Purpose Materials, ASM Handbook, ASM International 2, (1990) p. 542

[72] Tsutomu Tanaka, , Koichi Makiib, Hiroki Uedac, Atsumichi Kushibed, Masahide Kohzua, Kenji Higashia, "Study on practical application of a new seismic damper using a Zn–Al alloy with a nanocrystalline microstructure", Int. J Mech. Sci 45 Issue 10 (2003) 1599–1612.

[73] S. M. Lee and T. G. Langdon, "High Strain Rate Superplasticity in a Zn - 22% Al Alloy after Equal-Channel Angular Pressing", Mater. Sci. Forum. 357–359 (2001) 321– 326.

[74] M. Furukawa, Y. Ma, Z. Horita, M. Nemoto, R. Z. Valiev and T. G. Langdon, "Microstructural characteristics and superplastic ductility in a Zn-22% Al alloy with submicrometer grain size", Mater. Sci. Eng. A. 241 (1998) 122–128.

[75] Y. Kaneko, A. Vinogradov, K. Kitagawa and S. Hashimoto, "Cyclic Stress-Strain Response of Pb-Sn and Zn-Al Eutectic Alloys Fine-Grained by Equal Channel Angular Pressing", Investigations and Applications of Severe Plastic Deformation, Edited by Terry C. Lowe, Ruslan Z. Valiev (2000) 289–295.

[76] M.E. Cetin, M. Demirtas, H. Sofuoglu, O.N. Cora, G. Purcek, "Effect of grain size on room temperature deformation behavior of Zn-22Al alloy under uniaxial and biaxial conditions", Mater. Sci. Eng A 672 (2016) 78-87.

[77] M. Demirtas, G. Purcek, H. Yanar, Z. Zhang, Z.F. Zhang, “Effect of natural aging on RT and HSR superplasticity of ultrafine grained Zn-22Al alloy”, Mater. Sci. Forum, 838-839 (2016) 320-325.

[78] T. Tanaka, H. Watanabe and K. Higashi, “Microstructure in Zn-22Al Alloys after Equal-Channel-Angular Extrusion”, Mater. Trans. 44 No.9 (2003) 1891-1894.

[79] T. Nishihara, “Friction stir welding of superplastic Zn–22Al Alloy,” Proceedings of the Fourteenth International Offshore and Polar Engineering Conference 4 (2004) 78–82.

[80] P. P. Adrian, B. M. Gheorghe, “Manufacturing Process and Applications of Composite Materials”, Fascicle of Management and Technological Engineering, IX (XIX) (2010) NR2, 3.1-3.6.

[81] William D. Callister, Jr., “Materials Science and Engineering, An Introduction”, York, PA: John Wiley and sons, Inc. (2007) 578.

[82] Y. Nishida, “Introduction to Metal Matrix Composites: Fabrication and processing”, published by springer Japan (2013) 2.

[83] Saman Sahraeinejad, “Fabrication of Surface Metal Matrix Composites Using Friction Stir Processing”, thesis presented to the University of Waterloo (2014) 4-11.

[84] M. Rosso, "Ceramic and metal matrix composites: Routes and properties," Materials Processing Technology 175 (2006) pp. 364–375.

[85] William D. Callister, Jr., “Materials Science and Engineering, An Introduction”, York, PA: John Wiley and sons, Inc. (2007) 604.

[86] C. R. Kennedy, "Proceedings of the 7th CIMTEC-World Ceramics Congress," in Elsevier, New York (1991) 691.

[87] C. K. Kumar, Composite materials: science and engineering. New York: Springer, (2012) 168-170.

[88] A. J. Cook and P. S. Werner, "Pressure infiltration casting of metal matrix composites," Materials Science and Engineering: A. 144 (1991) 189-206.

[89] R. Saha, E. Morris, N. Chawla, and S. M. Pickard, "Hybrid and Conventional Particle Reinforced Metal Matrix Composites by Squeeze Infiltration Casting," Mater. Sci. Lett 21 (2002) 337-339.

[90] S. Nourbakhsh, F. L. Liang, and H. Margolin, "Interaction of Al2O3-ZrO2 fibres with a Ti-Al matrix during pressure casting," Metallurgical Transactions A. 21A (1990) 213–219.

[91] K. K. Chawla and L. B. Godfroid, "Fracture behaviour of Al 1100/Al 2024 laminate Composites" in Proceedings of the 6th International Conference on Fracture (ICF6) (1984, India).

[92] W. H. Hunt, "Processing and Fabrication of Advanced Materials," in the Minerals and Metal Materials Society, Warrendale, Pa (1994) 663−683.

[93] N. Chawla, J. J. Williams, and R. Saha, J. Light Metals. 2 (2002) 215-227.

[94] K. U. Kainer, "Metal Matrix Composites: Custom-made Materials for Automotive and Aerospace Engineering", Weinheim: WILEY-VCH Verlag GmbH and Co. KGaA (2006) 4.

[95] M. Mabuchi, T. Imai, "Superplasticity of Si3N4 Whisker Reinforced 6061 Aluminum at High Strain Rate", Journal of Materials Science Letters 9 (1990) 761-762.

[96] S. Kojima, T. Ishikawa, "Production of Superplastic Aluminium Sheet Clad With Metal Matrix Composite by Hot Rolling", Journal of the JSTP 47, No. 548 (2006) 47-52.

[97] H. Nishimura, H. Yamamoto, S. Yamagishi, "Fabrication of superplastic Zn-22Al alloy matrix composite", J. Japan Inst. of Light Metals 38 No 10 (1988) 665-671.

[98] Mahoney, M.W. & Ghosh, A.K. MTA, "Superplasticity in a high strength powder aluminum", Metall. Trans 18A (1987) 653-661.

[99] J. Pilling, "Superplasticity in aluminium base metal matrix composites", Scripta Metall 13 (1989) 1375-1380.

[100] H. Xiaoxu, L. Qing, C. K. Yao and Y. Mei, "Superplasticity in a SiC-6061Al composite", J. Mater. Sci. Lett 10 (1990) 964-966.

[101] Mabuchi, M., Imai, T. & Higashi, K., "Production of superplastic aluminium composites reinforced with Si3N4 by powder metallurgy", J. Mater. Sci. 28 (1993) 6582-6586.

[102] T. G. Nieh, C. A. Henshall, J. Wadswoth, Scripta Metal. 18 (1984) 1405.

[103] M. W. Mahoney, A. K. Ghosh, Metal. Trans 18A (1987) 653.

[104] G. G. Doncel at al., Compo. Sci. Tech. 35 (1989) 105.

[105] M. W. Mahoney, A. K. Ghosh, C. C. Bampton, ICCM 6 and ECCM 2 (London, 1987), vol. 2, 372.

[106] Idem, ibid. ASMI, Materials Park, OH, 1991) 275-282.

[107] E. D. Russo et al., Mat Sci. Eng. 14 (1974) 23.

[108] A. A. Baker, Mater. Sci, Eng. 17 (1975) 177-208.

[109] D. A. Hutto et al., Proceeding of 31st Int. SAMPE Symposium and Exhibition (1986) 145-153.

[110] C. H. Hamilton, L. A. Ascani, "Method for Superplastic Forming of Metals with Concurrent Diffusion Bonding (Nov 1975) US Patent 3920175.

[111] A. Velicki, "Method of Making a SPF/DB Hollow Core Fan Blade", (1995) US Patent number: 5,384,959.

[112] L. Ceschini, "Superplastic Forming (SPF) of Materials and SPF Combined with Diffusion Bonding Technological and Design Aspects", Journal of Metallurgical science and technology 10, No.3 (1992) 41-55.

[113] HEFTI Larry D., "Fine-Grain Titanium 6Al-4V for Superplastic Forming and Diffusion Bonding of Aerospace Products", JOM, Vol.62 No.5 (2010) 42-45.

[114] Du Zhihao, Jiang Shaosong, Zhang Kaifeng, Lu Zhen, "The structural design and superplastic forming/diffusion bonding of Ti2AlNb based alloy for four-layer structure", Materials & Design 104 (2016) 242-250.

[115] M. Mitsuji, Y. Ichige, S. Yaita, "Superplastic forming and diffusion bonding of aluminum alloys in various atmosphere", The Japan Society for Technology and Plasticity, Spring Lecture Conference Papers (1995) 399-400.

[116] T. Nagano, F. Wakai, "Superplastic Diffusion Bonding in Ceramics", Journal of the Ceramic Society of Japan 100, No.1167 (1992) 1279-1284.

[117] A. Azujima, "Composite vibration-damping laminated steel sheet for high-temperature use", J. Japan Inst. Met. Mater. 61, No.6 (1991) 46-52

[118] J. Kaneko, M. Sugamata, J. S. KIM, "Fabrication and Mechanical Properties of Zn-22 mass%Al Superplastic Alloy Composites Reinforced by SiC", J. Japan Inst. Met. Mater. 55, No.9 (1991) 986-993.

[119] S. Kim, M. Kazumata, J. Kaneko, "High temperature formability of sic-whisker-reinforced Zn-22Al alloy composite material", The Japan Society for Technology and Plasticity, Spring Lecture Conference Papers (1991) 283-286

[120] T. Uno, O. Aizawa, T. Nishihara, K. Wakabayashi, "Application of Composite of Zn-22Al Superplastic Alloy and Steel to Cylinder Head Gasket", The Japan Society for Heat Treatment 36, No.6 (1996) 403-407.

[121] Murr L. E., Trillo E. A., Li Y. Flores R. D., Nowak B. M., McClure J. C., "SolidState Flow Associated with the Friction-Stir Welding of Dissimilar Metals", The minerals, Metals & Materilas Society (1999) 31.

[122] Li Y, Murr L. E., McClure J. C., "Flow visualization and residual microstructures associated with the friction-stir welding of 2024 aluminum to 6061 aluminum", Materials Science and Engineering. A 271 (1999) 213-223.

[123] Y. Motohashi," Friction Stir Processing for Structure and Material Development and Applied technology", Journal of the Japan Society for Technology and Plasticity 50 No. 578 (2009) 2-6. (In Japanese)

[124] N. Saito, I Shigematsu, "Friction stir processing—A new technique for microstructure control of metallic materials", Journal of Japan Institute of Light Metals 57, No. 11 (2007) 492‐498. (In Japanese)

[125] Mishra R. S., "Friction stir processing for superplasticity", Advanced Materials and Processes (2004) 45-46.

[126] Mahoney M., Mishra R.S., Nelson T., Flintoff J., Islamgaliev R., Hovansky Y. (2001). "High strain rate, thick section superplasticity created via friction stir processing," Friction Stir Welding and Processing, Edited by K.V. Jata, M.W. Mahoney, R.S. Mishra, S.L. Semiatin, and D.P. Fields, The Minerals, Metals & Materials Society, 183–194.

[127] Mahoney M.W., Lynch S.P., "Friction stir processing," (2006) OMB, report No. 0704-0188.

[128] R. S. Mishra, Z. Y. Ma, I. Charit, "Friction stir processing: a novel technique for fabrication of surface composite", Mater. Sci. Eng. A 341 (2003) 307-310.

[129] J. Lee, J. C. Huang, P. J. Hsieh, "Mg based nano-composites fabricated by friction stir processing", Scr. Mater. 54 (2006) 1415-1420.

[130] D. K. Lim, T. Shibayanagi, A. P. Gelich, “Synthesis of multi-walled CNT reinforced aluminium alloy composite via friction stir processing”, Mater. Sci. Eng. A 507 (2009) 194-199.

[131] M. Dixit, J. W. Newkirk, R. S. Mishra, “Properties of friction stir-processed Al 1100–NiTi composite”, Scr. Mater. 56 (2007) 541-544.

[132] A. Shafiei-Zharghani, S. F. Kashani-Bozorg, A. Zarei-Hanzaki, “Microstructures and mechanical properties of Al/Al2O3 surface nano-composite layer produced by friction stir processing”, Mater. Sci. Eng. A 500 (2009) 84-91.

[133] P. Asadi, G. Faraji, A. Masoumi, M. K. Besharati Givi, “Experimental Investigation of Magnesium-Base Nanocomposite Produced by Friction Stir Processing: Effects of Particle Types and Number of Friction Stir Processing Passes”, Metal. Mater. Trans. A 42 Isshue 9 (2011) 2820-2832.

[134] Y. Hangai, T. Utsunomiya, “Manufacture of Porous Aluminum by Utilizing Friction Stir Processing”, J. Japan Inst. Met. Mater. 73-2 (2009) 131-133.

[135] K. Yamamura, T. Nishihara, “Development of Local Reinforcement and Local Metallic Foam using FSP”, Mater. Sci. Forum 638-642 (2010) 1267-1272.

[136] T. Morooka, E. Yuasa, T. Shu, “Preparation of fine steel wire-reinforced aluminium composite by the powder rolling”, J. Japan Inst. Light Metals 37 No 3 (1987) 199-206.

[137] M. Furukawa, Y. Ma, Z. Horita, M. Nemoto, R. Z. Valiev and T. G. Langdon, “Microstructural characteristics and superplastic ductility in a Zn-22%Al alloy with submicrometer grain size”, Mater. Sci. Eng. A, 241 (1998) 122-128.

[138] S.M. Lee and T. G. Langdon, ”High Strain Rate Superplasticity in a Zn-22%Al Alloy after Equal-Channel Angular Pressing”, Mater. Sci. Forum 357-359 (2001) 321-326.

[139] R. S. Mishra, M. W. Mahoney, “Friction Stir Processing: A New Grain Refinement Technique to Achieve High Strain Rate Superplasticity in Commercial Alloys”, Mater. Sci. Forum, 357-359 (2001) 507-514.

[140] J. Q. Su, T. W. Nelson, C. J. Sterling, “Grain Refinement of Aluminum Alloys by Friction Stir Processing”, Philosophical Magazine 86, Issue 1 (2006) 1-24.

[141] K. Yamamura, N. Kawada, T. Nishihara, “Formation of Local Metallic Foam of Aluminum Alloy Using Friction Stir Processing”, J. Japan Inst. Metals 74, No. 1 (2010) 55-60.

[142] M. Mabuchi, T. Imai, "Production of superplastic aluminium composites reinforced with Si3N 4 by powder metallurgy ", J. Mater. Sci. (1993), 6582-6586.

[143] Mahoney M., Mishra R.S., Nelson T., Flintoff J., Islamgaliev R., Hovansky Y. (2001). “High strain rate, thick section superplasticity created via friction stir processing,” Friction Stir Welding and Processing, Edited by K.V. Jata, M.W. Mahoney, R.S. Mishra, S.L. Semiatin, and D.P. Fields, The Minerals, Metals & Materials Society, 183–194.

[144] Mahoney M.W., Lynch S.P. (2006). “Friction stir processing,” OMB, report No. 0704-0188.

[145] T. G. Nieh, J. Wadsworth and O. D. Sherby, Superplasticity in Metals and Ceramics, Cambridge University Press, Cambridge, UK (1997) 91–124.

[146] Hamilton C.H., Ghosh, A.K. (1988). “Superplastic sheet forming,” Metals Handbook 9th Edition, ASM International, 14, 852.

[147] Nishihara T. (2004). “Friction stir welding of superplastic Zn–22Al Alloy,” Proceedings of the Fourteenth International Offshore and Polar Engineering Conference, 4, 78–82.

[148] T. Tanaka, K. Makiib, H. Uedac, A. Kushibed, M. Kohzua, K. Higashia, “Study on Practical application of a new seimic damper using a Zn-Al alloy with a nanocrystalline microstructure”, Int. J. Mech. Sci. 45 (2003) 1599-1612.

[149] Hall E.O., “The deformation and ageing of mild steel: III Discussion of results,” Proceedings of the Physical Society 64 (1951) 747–753.

[150] Petch N.J., “The cleavage strength of polycrystals,” J. Iron and Steel Inst. 174 (1953) 25–28.

[151] Ashby M.F., Jones D.R.H., Engineering Materials 1: An Introduction to their Properties and Applications, Pergamon Press, Oxford (1980) 105.

[152] Sato Y.S., Urata M., Kokawa H., Ikeda K., “Hall–Petch relationship in friction stir welds of equal channel angular-pressed aluminum alloys,” Mater. Sci. Eng. A 354 (2003) 298–305.

[153] Suresh D. Meshram, G. Madhusudhan Reddy, A. Venugopal Rao, "Role of Threaded Tool Pin Profile and Rotational Speed on Generation of Defect Free Friction Stir AA 2014 Aluminium Alloy Welds", Defence Science Journal 66, No. 1 (2016) 57-63.

[154] P. Satish Kumar, Ch.S.R.Sastry, A.Devaraju, M.Shiva Chander, "Effect of Taper with Threaded Tool Profile on Mechanical and Microstructural Properties of Friction Stir Welded 5083 Aluminum Alloy", International Journal of Research in Engineering and Technology 5 Issue 7 (2016) 224-228.

[155] A. Ikuta, H. Kyogoku, T. H. North, "Material Flow by Tools for Friction Stir Spot Welding", Kinki University Next Generation Fundamental Technology Laboratory Report 4 (2013) 55-61.

[156] A. Hanai, A. Shimizu, H. Tsumoto, H. Yokoyama, T. Sugimoto, Y. Yoshida, "Effect of Tool Tip Shape on the Mechanical Properties of Friction Stir Spot Welds", Aichi Industrial Science and Technology Center Research Report (2015) 18-21.

[157] Y. N. Zhang, X. Cao, S. Larose, P. Wanjara, "Review of tools for friction stir welding and processing", Canadian Metallurgical Quarterly 41, No. 3 (2012) 251-261.

[158] R. S. Mishra and M. W. Mahoney, "Friction stir welding and processing", Materials Park, OH, ASM International (2007).

[159] L. Dubourg and P. Dacheux, "Design and properties of FSW tools: a literature review", Proc. 6th Int. Symp. on Friction stir welding 52, No. 4, (2006) 62.

[160] P. A. Colegrove and H. R. Shercliff, "Development of trivex friction stir welding tool. Part 1: two-dimensional flow modelling", Sci. Technol. Weld. Join. 9, No. 4 (2004) 345–351.

[161] P. A. Colegrove and H. R. Shercliff, "Experimental and numerical analysis of aluminum alloy 7075-T7351 friction stir welds", Sci. Technol. Weld. Join. 8, No. 5 (2003), 360–368.

[162] Tang W., Guo X., McClure J.C., Murr L.E. (1998). "Heat input and temperature distribution in friction stir welding," J. Mater. Process. Manu. Sci. 7 (1998) 163–172.

[163] Frigaard Ø., Grong Ø., Bjorneklett B., Midling O.T. (1999). “Modelling of the thermal and microstructural fields during friction stir welding of aluminum alloys,” Proceedings of the 1st International Symposium on Friction Stir Welding, Thousand Oaks, CA, USA, CD-ROM.

[164] D. Storjohann, O. M. Barabash, S. A. David, P. S. Sklad, E. E. Bloom, S. S. Babu, “Fusion and Friction Stir Welding of Aluminum-Metal-Matrix Composites,” Metall and Mat Trans A 36 (2005) 3237-3247.

[165] K. Yamamura, and T. Nishihara, “Friction Stir Welding of SiC Whisker Reinforced Aluminum Composites” Paper presented at ISOPE2007, Lisbon, Portugal (2007) 483.

[166] R.S Mishra and Z.Y.Ma “Friction stir welding and processing” Mater. Sci. Eng. R. Reports 50, No 1-2 (2005) 1-78.

[167] H Mofidi, T Nishihara “Effect of friction stir processing on superplastic behavior of Zn-22Al alloy”, proceedings of the 1st International Joint Symposium on Joining and Welding, Osaka, Japan (2013) 383-387.

[168] W. Tang, X. Guo, J.C. McClure, L.E. Murr, “Heat Input and Temperature Distribution in Friction Stir Welding”, J. mater. Proceed. Manu. sci. (1998) 163-172.

[169] C.P. Wang, “Recent Advance in Hermetic Equivalent Flip-chip Hybrid IC Packaging of Microelectronics”, Mater. Chem. Phys. 42 (1995) 25-30.

[170] C.T. Nguyen, “Micromachining technologies for miniaturized communication devices”, Proc. SPIE 3514 (1998) 24-38.

[171] K. Najafi, “Silicon micromachining technologies: Future needs and challenges”, Proc. SPIE 2879 (1996) 206-215.

[172] S.T. Chou and F. M. Erdamann, “An On-chip Hermetic Package Technology for Micromechanical Devices”, Proc. Tech. Dig. Solid State Sensor and Actuator Workshop. USA, (1998) 229-232.

[173] D. Ando, K. Oishi, T. Nakamura, and S. Umeda, “Glass direct bonding technology for hermetic seal package”, Proc. IEEE MicroElectro Mechanical Systems. IEEE. Japan, Nagoya. (1997) 186-190.

[174] B. Shivkumar and C. J. Kim, “Microrivets for MEMS packaging: Concept, fabrication, and strength testing”, J. Microelectromech. Syst. 6 (1997) 217-225.

[175] M.B. Cohn, K.F. Bohringer, J.M. Noworolski, A. Singh, and C. G. Keller, K.Y. Goldberg, and R.T. Howe, "Microassembly technologies for MEMS", Proc. SPIE 3512 (1998) 2-16.

[176] M. Peel, A. Steuwer, M. Preuss, and P.J. Withers, "Microstructure, Mechanical Properties and Residual Stresses as a Function of Welding Speed in Aluminum AA5083 Friction Stir Welds", Act. Mater. 51 (2003) 4791-4801.

[177] F. Gratecap, G. Racineux, and S. Marya "A Simple Methodology to Define Conical Tool Geometry and Welding Parameters in Friction Stir Welding", 7th International Friction Stir Welding Symp TWI, Japan (2008) published on CD.

[178] A. Simar, Y. Brechet, B. de Meester, A. Denquin, and T. Pardon "Microstructure, local and global mechanical properties of friction stir welds in aluminum alloy 6005A-T6", Mater. Sci. Eng. A 486 No. 1-2 (2008) 85-95.

[179] R.S. Mishra "Friction Stir Processing for Superplasticity", Adv. Mater. Process. 162 No 2 (2004) 45-47.

[180] H. Mofidi and T. Nishihara "Effect of friction stir processing on superplastic behavior of Zn-22Al alloy", Proc. 1st Int. Joint Symp. on Joining and Welding ed. by H. Fujii Joining and Welding Inst. Japan (2013) 383-387.

[181] W. Tang, X. Guo, J.C. McClure, and L.E. Murr "Heat Input and Temperature Distribution in Friction Stir Welding", J. Mater. Process. Manuf. Sci. 7 (1998) 163-172.

[182] N. Balasubramanian, R.S. Mishra, and K. Krishnamurthy "Process force during friction stir channeling in an aluminum alloy", J. Mater. Process. Technol. 211 (2011) 305-311.

[183] Y.S. Sato, H. Takauchi, S.H.C. Park, and H. Kokawa "Characteristics of the kissing-bond in friction stir welded Al alloy 1050", Mater. Sci. Eng. A 405 (2005) 333-338.

[184] H. Mofidi and T. Nishihara, "Production of a Superplastic Vibration-Damping Steel Sheet Composite using Friction Stir Forming", Mater. Sci. Forum 838-839 (2016) 574-580.

[185] J. Beddoes, W. Wallace, and L. Zhao, "Current understanding of creep behaviour of near γ-titanium aluminides", Int. Mater. Rev. 40 (1995) 197-217.

[186] H.Y. Kim and K. Maruyama, “Stability of lamellar microstructure of hard orientated PST crystal of TiAl alloy”, Act. Mater. 51 (2003) 2191-2204.

[187] K. Maruyama, M. Yamaguchi, G. Suzuki, H. Zhu, H.Y. Kim, and M. H. Yoo, “Effects of lamellar boundary structural change on lamellar size hardening in TiAl alloy”, Act. Mater. 52 (2004) 5185-5194.

[188] S.N. Patankar, Y.T. Kwang, and T.M. Je, “Alpha casing and superplastic behavior of Ti–6Al–4V”, J. Mater. Proc. Technol. 112 (2001) 24-28.

[189] S. Rhaipu, “The effect of microstructural gradients on superplastic forming of Ti–6Al–4V”, J. Mater. Proc. Technol. 80-81 (1998) 90-95.

[190] Y.H. Kim, J.-M. Lee, and S.S. Hong, “Optimal design of superplastic forming processes”, J. Mater. Proc. Technol. 112 (2001) 166-173.

[191] J.R. Davis 2001 ASM Specialty Handbook. Copper and Copper alloys. ASM International. USA. p. 35.

[192] Available at: http://www.yamanaka.co.jp/function/naslon003.html

[193] M W. Mahoney, W. Harrigan and JA. Wert, “Joint in Aluminum” INALCO’98 (1998) 261-270.

[194] T. Hara, T. Nishihara, “Friction Stir Lap-Welding of Zn-22Al Superplastic Alloy", Japan Society of Mechanical Engineering, M&P 2012, on CD.

PUBLICATIONS *(Peer reviewed)*

1) **H. Mofidi,** T. Hara, T. Nishihara, *"Production of a Superplastic Vibration-Damping Steel Sheet Composite using Friction Stir Forming"*, Materials Science Forum, ISSN: 1662-9752, Vol. 838-839 (2016), pp 574-580.

2) **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Friction Stir Forming for Mechanical Interlocking of Insulated Copper Wire and Zn-22Al Superplastic Alloy"*, Welding in the World, (The International Journal of Materials Joining), ISSN: 0043-2288, Vol. 61, Issue 1 (2017), pp, 44-55.

3) **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Grain Refinement of Zn-22Al Superplastic Alloy Using Friction Stir Processing"*, IJOPE (International Journal of Offshore and Polar Engineering), ISSN: 1053-5381, Vol. 27, No.2 (2017) pp 204-209.

4) Takahiro Ohashi, Jiazhao Chen, Tadashi Nishihara, **Hamed Mofidi Tabatabaei**, *"Friction Stir Forming of Aluminum Alloy Gear-Racks"*, Key Engineering Materials, ISSN: 1662-9795, Vol. 725 (2016) pp 665-670.

5) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tetta Ikeya, Tadashi Nishihara, *"Friction Stir Forming of A5083 Aluminum Alloy Gear-Racks with WC Particles Embedded in Tooth Surface"*, Key Engineering Materials, ISSN: 1662-9795, Vol. 723 (2016) pp 148-153.

6) Takahiro Ohashi, **Hamed Mofidi Tabatabaei,** Tadashi Nishihara, *"Cylindrical Pin Embossment on A5083 Aluminum Alloy Substrate Fabricated by Friction Stir Forming"*, Key Engineering Materials, ISSN: 1662-9795, Vol. 730 (2016) pp 253-258.

7) Takahiro Ohashi, **Hamed Mofidi Tabatabaei** Tadashi Nishihara, *"Observation of Material Flow in Friction Stir Forming for A5083 Aluminum Alloy Gear-Rack"*, Materials Science Forum, ISSN: 1662-9752, Vol. 889 (2016) pp 113-118.

8) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Low Height Ultra-Thin Fin on A5083 Aluminum Plate Fabricated by Friction-Stir Forming"*, Procedia Engineering Vol. 174 (2017) 74 – 81.

9) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Vertical Wall on an Aluminum Alloy Plate Fabricated"*, Key Engineering Materials, ISSN: 1662-9795, Vol. 748 (2017) pp 202-206.

10) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Fastenerless-Riveting Utilizing Friction Stir Forming for Dissimilar Materials Joining"*, Key Engineering Materials, ISSN: 1662-9795, Vol. 751 (2017) pp 186-191.

11) Takahiro Ohashia, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Rib-Structures on A5083 Aluminum Alloy Sheet Generated by Friction Stir Forming"*, Procedia Engineering, Vol. 207 (2017) pp 1153–1158.

12) **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Friction Stir Forming for Mechanical Interlocking of an Ultra-thin Stainless Steel Strands with Aluminum Alloys"*, Defect and Diffusion Forum, Vol. 382 (2018), pp 114-119.

13) Takahiro Ohashi, Xin Tong1, Zijie Zhao1, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, "Evaluation of Material Deformability and Pressure Distribution on a Die Surface under a Tool in Spot FSF", Defect and Diffusion Forum, Vol. 382 (2018), pp 132-137.

14) **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, "Effect of Friction Stir Welding on Mechanical Properties of Zn-22Al Superplastic Alloy", International Journal of Engineering Research in Mechanical and Civil Engineering, ISSN (Online) 2456-1290, Vol 2, Issue 12 (2017) pp. 63-68.

International Conferences

1) **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, "*Effect of friction stir processing on superplastic behavior of Zn-22Al alloy*", proceedings of the 1st International Joint Symposium on Joining and Welding, 2013, Edited by H. Fuji, Woodhead Publishing, ISBN 978-1-78242-163-4, pp 383-387.

2) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, and Tadashi Nishihara, "*Mechanical Behavior and Failure of Easily-Decomposable Dissimilar-Materials-joint Fabricated by Friction Stir Forming"*, The 6th International Conference on Materials and Processing, JSME/ASME ICM&P, 2017, Los Angeles, June 4-8.

3) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, and Tadashi Nishihara, *"Cylindrical Extrusions on A5083 Aluminum Alloy Plate Fabricated by Friction Stir Forming"*, 20th International ESAFORM Conference on Material Forming April 26-28, 2017, Ireland, AIP Conference Proceedings 1896, 080002.

4) **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, "Effect of Friction Stir Welding on Mechanical Properties of Zn-22Al Superplastic Alloy", International Conference on Researches in Science and Technology, ICRST- 17, Dec 23-24, 2017, Tehran, ISBN: 9788192958045, pp 48-53.

Conference papers in Japan

1) **Hamed Mofidi** , Tadashi Nishihara, *"Control of Zn-22Al Superplastic Alloy Microstructure Using Friction Stir Processing"*, The Japan Society of Mechanical Engineers, Proceedings of the 18th Materials and processing technical conference, Tokyo [M&P (2010)] Published on CD.

2) **Hamed Mofidi** , Tadashi Nishihara, *"Effect of Friction Stir Processing on Mechanical Behavior of Zn-22Al Superplastic Alloy"*, The Japan Society of Mechanical Engineers, Mechanical Engineering Congress, Kanazawa (2012) (MECJ-12) Published on CD.

3) **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Mechanical Interlock of an Insulated Copper Wire with Zn-22Al Superplastic Alloy Using Friction Stir Forming",* The Japan Society for Technology of Plasticity, The Proceedings of the 67th Japanese Joint Conference for the Technology of Plasticity, Saitama (2016) pp. 159-160.

4) **Hamed Mofidi Tabatabaei** , Tadashi Nishihara, *"Trials of Mechanical Interlocking of Aluminum Alloys with Stainless Steel Strands Using Friction Stir Forming"*, The Japan Institute of Light Metals, The 132th Conference of Japan Institute of Light Metals Nagoya (2017) pp.279-280.

5) **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Mechanical Interlocking of Aluminum Alloys with Stainless Steel Strands Using Friction Stir Forming"* ,The Japan Society for Technology of Plasticity, The Proceedings of the 2017 Japanese Spring Conference for the Technology of Plasticity, Gifu (2017). pp. 283-284.

6) Shigeru Okada, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, Takashi Yamazaki, Kunio Yubuta, Toetsu Shishido, Akiko Nomura (Materials Organization) Takao Mori, *"Synthesis of ternary boride crystals and their effect on hardness"*, Journal of Flux Growth ISSN 1881-5316, Vol. 10, No.2 (2015) pp.121-122

7) Takashi Yamasaki, Yuta Suzuki, Shigeru Okada, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, Kunio Yubuta, Akiko Nomura, Toetsu Shishido, Takao Mori, *"Crystal synthesis and properties of chromium silicide using Ga flux"*, The Ceramic Society of Japan, Annual Meeting 2016, Published on DVD.

8) Takahiro Ohashi, Jiazhao Chen ,Tadashi Nishihara and **Hamed Mofidi Tabatabaei**, *"Net Shape Forming of a Gear Rack Utilizing Friction Stir Forming"*, The Japan Society for Technology of Plasticity, The Proceedings of the 2016 Japanese Spring Conference for the Technology of Plasticity, Kyoto (2016) pp. 205-206.

9) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Proposal of Fastenerless-Riveting Utilizing Friction Stir Forming"*, The Japan Society for Technology of Plasticity, The Proceedings of the 67th Japanese Joint Conference for the Technology of Plasticity, Saitama (2016), pp. 167-168.

10) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Observation of Metal flow during Friction Stir Forming of a Gear-Rack"*, The Japan Society for Technology of Plasticity, The Proceedings of the 67th Japanese Joint Conference for the Technology of Plasticity, Saitama, (2016), pp. 325-326.

11) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Proposal of Reinforcement of Sheet Shaped Products by the Rib-Structure Generated by Friction Stir Forming (FSF)"*, The Japan Society for Technology of Plasticity, The Proceedings of the 67th Japanese Joint Conference for the Technology of Plasticity, Saitama, (2016), pp. 327-328.

12) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, "Embodiments of the Friction Stir Forming (FSF) Approach", The Japan Society of Mechanical Engineers, Innovation from JSME 2016

13) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Easily-Decomposable Dissimilar-Materials-Joining with Employing Friction Stir Forming (FSF)"*, The Japan Society of Mechanical Engineers, Proceedings of the 24th Materials and processing technical conference, Tokyo [M&P (2016)] Published on USB.

14) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"6. Cylindrical Pin Embossment on A5083 Aluminum Alloy Substrate Fabricated by Friction Stir Forming"*, The Japan Society of Mechanical Engineers, Proceedings of the 24th

Materials and processing technical conference, Tokyo [M&P (2016)] Published on USB.

15) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Joining Of CFRP and Aluminum Alloy Plate Employing Friction Stir Forming (FSF)"*, The Japan Society of Mechanical Engineers, Proceedings of the 24th Materials and processing technical conference, Tokyo [M&P (2016)] Published on USB.

16) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Generation of low-profile thin plate-fins on A5083 medium gauge plate by friction stir forming (FSF)"*, The Japan Institute of Light Metals, The 132th Conference of Japan Institute of Light Metals Nagoya (2017), pp 277-278.

17) Takahiro Ohashi, **Hamed Mofidi Tabatabaei**, Tadashi Nishihara, *"Strength Evaluation of Joints Between Dissimilar Materials Formed by Friction Stir Forming (FSF)"*, The Japan Society for Technology of Plasticity, The Proceedings of the 2016 Japanese Spring Conference for the Technology of Plasticity, Gifu (2017). pp. 125-126.