

**STUDIES ON THE PERFORMANCE OF MILD STEEL-ALUMINA JOINING  
FABRICATED VIA FRICTION WELDING**

**by**

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## LIST OF SYMBOLS

$\lambda$  Wave length

$\text{\AA}$  angstrom

$\theta$  Bragg angle

## LIST OF ABBREVIATIONS

Ag	Argentums
Al	Aluminium
Cr	Chromium
Cu	Cooper
Fe	Ferum
Hf	Hafnium
Mn	Manganese
Mo	Molybdenum
Nb	Neobium
Ni	Nickel
Ta	Tantalum
Th	Thorium
Ti	Titanium
V	Vanadium
Zr	Zirconium
Al <sub>2</sub> O <sub>3</sub>	Alumina
SiC	Silicon carbide
Si <sub>3</sub> N <sub>4</sub>	Silicon nitride
TiAl	Titanium aluminide
TiN	Titanium nitride
TiO	Titanium oxide
Ti <sub>2</sub> O <sub>3</sub>	Titanium Dioxide
ZrO <sub>2</sub>	Zirconium oxide
ZTM	Zirconium oxide toughened mullite
PSZ	Partially stabilize zirconium
TLPB	Transient liquid partial braze
PTLPB	Transient liquid partial braze
HAZ	Heat affected zone

$T_m$	Melting point
R	Radius
$R_w$	New radius
SFW	Stir Friction Welding
LFW	Linear Friction Welding
RFW	Radial Friction Welding
TWI	The Welding Institute
AISI	American Iron and Steel Institute
ASTM	American standard testing and measurement
EDS	Energy dispersive spectrometer
EDX	Energy Dispersive X-ray
ICDD	International Centre of Diffraction Data
FESEM	Field emission scanning electron microscopy
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
POP	Plaster of Paris
MPa	Mega Pascal
rpm	Revolution per minute
KHN	Knoop hardness number
ITEX	International Technology and Engineering Exhibition

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## **LIST OF PUBLICATIONS**

- 1.1 Mohamad Zaky Noh, Yeoh Cheow Keat, Luay Bakir Hussain, Zainal Arifin Ahmad. (2004). A Study on the using Al Interlayer on Ceramics-Metals Bonding through Friction Welding Technique. Poster Presentation at 2<sup>nd</sup> National Postgraduate Colloquium on Materials, Minerals and Polymers, 7 – 8<sup>th</sup> October 2004
- 1.2 Mohamad Zaky Noh, Yeoh Cheow Keat, Luay Bakir Hussain, Zainal Arifin Ahmad. (2005). Effect of Interlayer Thickness on Alumina-Steel Bonding Through Friction Welding. Malaysian Journal of Microscopy. 1: 94 – 99.
- 1.3 Mohamad Zaky Noh, Luay Bakir Hussain, Zainal Arifin Ahmad. (2005) Effect of Friction Time on the Strength of Alumina-Steel Bonding Through Friction Welding. Proceedings of Brunei International Conference on Engineering & Technology, Institut Teknologi Brunei, Bandar Seri Begawan, Brunei Darussalam, 15 – 18<sup>th</sup> August 2005. 91 – 97.
- 1.4 Mohamad Zaky Noh, Luay Bakir Hussain, Zainal Arifin Ahmad. (2006). The Study of Speed Rotation on the Strength of Steel-Alumina Friction Welding. Malaysian Journal of Microscopy. 1: 98 – 103.
- 1.5 Mohamad Zaky Noh, Luay Bakir Hussain, Zainal Arifin Ahmad. Aluminum Sheet as Interlayer for Ceramic-Metal Joints via Friction Welding Technique. Oral Presentation at National Seminar on Science and Its Application in Industry, KUTKM, Melaka, 14-15<sup>th</sup> Feb 2006.
- 1.6 Mohamad Zaky Noh, Luay Bakir Hussain, Zainal Arifin Ahmad. Effect of Rod Diameter on the Strength of the Alumina-Steel Friction Bonded. Poster presentation at The Second International Conference on Solid State Science and Technology, KUSTEM, Terengganu, 4 – 6<sup>th</sup> Sept 2006.
- 1.7 Mohamad Zaky Noh, Luay Bakir Hussain, Zainal Arifin Ahmad. A microstructure Study on the  $\text{Al}_2\text{O}_3$ -Al and Al-Steel Interfaces for Ceramic-Metal Joints via Friction Welding. Poster Presentation at International Conference on X-Ray and Related Techniques in Research and Industry (ICXRI 2006), Palm Garden Hotel, Putrajaya, Malaysia, 29<sup>th</sup> - 30<sup>th</sup> November 2006.
- 1.8 Mohamad Zaky Noh, Luay Bakir Hussain, Zainal Arifin Ahmad. Correlation between the Bending Strength and the Thickness Interlayer of Alumina-Mild Steel Friction Welded at Lower Rotational Speed. Poster Presentation at 15<sup>th</sup> Scientific Conference and 16<sup>th</sup> Annual General Meeting, Electron Microscopy Society of Malaysia, Sutra Beach Resort, Kuala Terengganu, Malaysia, 4<sup>th</sup> - 6<sup>th</sup> December 2006.

- 1.9 Mohamad Zaky Noh, Luay Bakir Hussain, Zainal Arifin Ahmad. (2007). Alumina-Mild Steel Friction Welded at Lower Rotational Speed. Article in Press, Journal of Materials Processing Technology

## **STUDIES ON THE PERFORMANCE OF MILD STEEL-ALUMINA JOINING FABRICATED VIA FRICTION WELDING**

### **ABSTRACT**

Joining of metal-ceramic can be done by different techniques such as brazing, diffusion bonding and friction welding. The joint strength of metal and ceramic has been studied extensively due to the differences in their mechanical and chemical properties. However, the mechanism of metal-ceramic joining has not yet fully understood. In this study a 10 mm diameter of mild steel rods were friction welded with 10 mm diameter of alumina rods. Aluminum sheets with thickness of 0.3 mm, 0.5 mm and 1.5 mm were used as an interlayer. Friction and forging pressures of 20 MPa and 40 MPa were applied, respectively. The rotational speeds of 900, 1250, 1800 and 2500 rpm were used. The friction durations were varied accordingly from 2 s to 20 s with an interval of 2 s. Four points bending test was used to measure the strength of the joints. For the effect of friction times to the joint strength at various rotational speeds (900, 1250, 1800 and 2500 rpm), the bending strength is in the range of 50 – 190 MPa, 90 – 206 MPa, 100 – 195 MPa and 60 – 195 MPa, respectively were obtained. For the effect of interlayer thickness to the joint strength, it is obtained that the interlayer thickness of 1.0 mm produced the best joint strength for all the rotational speeds. The bending strength obtained is in the range of 130 – 190 MPa, 150 – 195 MPa, 150 – 180 MPa and 150 – 195 MPa, respectively. The optimum rotational speed to produce good joint strength for all interlayer thickness is 1250 rpm. The bending strength obtained is in the range of 120 – 150 MPa, 150 – 195 MPa and 150 – 205 MPa, respectively. Fracture surface analysis was carried out after the bending test. Microhardness and thermal

expansion tests were carried out on the joining sample. Most of the fracture occurred at the adjacent interlayer with small amount of alumina part attached to the aluminum interlayer. Microhardness test and thermal expansion show insignificant changes. By using optical microscope, the interlayer thickness was determined. The friction times, initial interlayer thickness and speed rotation significantly affected the average thicknesses of the interlayer. The average thickness interlayer of 100 to 200  $\mu\text{m}$  produced better joint strength. Deformation of the aluminum interface was clearly observed under FESEM. Mechanical interlocking and close contact between mild steel-aluminum and aluminum-alumina interfaces were observed at magnifications of 2000X. Therefore, the strength of alumina-mild steel bonding is much dependent on speed rotation, friction time and the thickness of aluminum interlayer. It is also much dependent on the wettability of the alumina surface by the partially molten aluminum interlayer and the existence of mechanical interlocking between interlayer and mild steel.

## **KAJIAN KE ATAS PRESTASI SAMBUNGAN BESI KELULI-ALUMINA YANG DIHASILKAN MELALUI KIMPALAN GESERAN**

### **ABSTRAK**

Sambungan seramik-keluli boleh dilakukan dengan menggunakan pelbagai teknik seperti pateri keras, ikatan resapan dan kimpalan geseran. Kajian tentang kekuatan sambungan keluli dan seramik telah banyak dilakukan kerana perbezaan sifat mekanikal dan kimianya. Walau bagaimana pun, mekanisma sambungan keluli-seramik ini masih belum difahami sepenuhnya. Dalam kajian ini rod keluli berdiameter 10 mm disambungkan dengan rod alumina berdiameter 10 mm secara kimpalan geseran. Kepingan aluminum berketalan 0.3 mm, 0.5 mm, 1.0 mm dan 1.5 mm digunakan sebagai bahan pengantara. Tekanan geseran dan tekanan tempaan yang digunakan ialah 20 MPa dan 40 MPa. Laju putaran iaitu 900, 1250, 1800 dan 2500 pusingan per minit telah digunakan. Tempoh geseran yang digunakan ialah dari 2 hingga 20 saat dengan selang masa selama 2 saat. Ujian lenturan empat titik digunakan untuk mengukur kekuatan sambungan. Untuk kesan masa geseran kepada kekuatan sambungan pada pelbagai laju putaran (900, 1250, 1800 dan 2500 ppm), didapati kekuatan lenturan adalah dalam julat 50 – 190 MPa, 90 – 206 MPa, 100 – 195 MPa dan 60 – 195 MPa. Untuk ketebalan pengantara kepada kekuatan sambungan, adalah didapati bahawa pengantara berketalan 1.0 mm menghasilkan kekuatan sambungan yang terbaik bagi semua laju putaran. Kekuatan lenturan didapati dalam julat 130 – 190 MPa, 150 – 195 MPa, 150 – 180 MPa dan 150 – 195 MPa. Laju putaran yang optimum untuk menghasilkan kekuatan sambungan yang baik untuk semua ketebalan pengantara ialah 1250 ppm. Kekuatan lenturan didapati masing-masing dalam julat 120 – 150 MPa,

150 – 195 MPa dan 150 – 205 MPa. Analisis permukaan patah dilakukan selepas ujian lenturan. Ujian kekerasan mikro dan pengembangan terma juga dilakukan ke atas sampel sambungan. Kebanyakan patah berlaku berdekatan pengantara dengan sebahagian kecil alumina terlekat pada pengantara aluminium. Ujian mikro kekerasan dan pengembangan terma menunjukkan tiada perubahan yang penting berlaku. Dengan menggunakan mikroskop optik, ketebalan pengantara ditentukan. Masa geseran, ketebalan awal pengantara dan laju putaran memberikan kesan yang penting kepada ketebalan purata pengantara selepas sambungan. Ketebalan purata pengantara di antara 100 hingga 200  $\mu\text{m}$  menghasilkan kekuatan sambungan yang baik. Ubahbentuk antaramuka aluminium dapat dilihat dengan jelas dengan menggunakan mesin pengimbas elektron. Kekunci mekanikal dan keadaan sentuhan yang rapat di antara keluli-aluminium dan aluminium-alumina dapat dilihat pada pembesaran 2000X. Kekuatan sambungan antara keluli-alumina banyak bergantung kepada laju putaran, masa geseran dan ketebalan pengantara aluminium. Kekuatan sambungan juga bergantung kepada keupayaan leburan aluminium membiasahi permukaan alumina dan kewujudan kekunci mekanikal antara bahan pengantara dan keluli.

## **CHAPTER 1 INTRODUCTION**

### **1.1 Metal-Ceramic Joining via Friction Welding**

The advancement of engineering and technology to meet future requirements in many diverse fields demands the development of new ceramic materials. No single ceramic material can meet all needs and consequently many different types of material require further investigation to meet that specific requirement. Ceramics and metals are the oldest established engineering materials. Ceramics have some attractive properties compared to metals and polymers, which make them useful for specific applications. Based on their physical, mechanical, thermal, electrical and chemical properties, ceramic materials have been utilized for many applications.

Structural ceramics have some superior properties compared to metals but they are difficult to machine due to their brittleness. In order to avoid the processing limitations, some form of joining is usually employed to form the final component. Therefore, ceramic/metal joints become more and more important in modern technology because of the unique combination of properties of metals like ductility, high electrical and thermal conductivity and the properties of ceramics like high hardness, high temperature strength, low thermal expansion, corrosion resistance and excellent wear resistance.

Friction welding is widely used for joining similar and dissimilar metals but for joining ceramics to metals is very challenging and requires very systematic experiments. Extensive efforts have been devoted to investigate the

appropriate processes to join dissimilar materials. There are some reports on the friction welding of ceramics to metals. Kanayama et al. (1985) carried out the experiments on the joining of two cylindrical bars and also tubes of 95 % alumina to each other using friction welding. Among the most extensive study was carried out by Essa and Bahrani (1991) which come out with the friction welding of aluminum alloy and alumina.

Ikeuchi et al. (1992) tried an attempt on the friction welding of ceramics to metals with the aid of intermediate layer of active metals. The ceramics and metals employed were a pressureless-sintered silicon carbide and a commercially pure nickel. Tsuchiya et al. (1994) studied the mechanical properties and the optimum fabricating condition of the alumina dispersed copper alloys to stainless steel joint which fabricated through friction welding. Furthermore, the influence of various parameters on the strength of friction welded ceramics-metals joint was examined by the finite element method (FEM) in combination with Weibull statistics [Weiss and Sassani, 1998].

Lin et al. (1999) investigated that the effect of joint design and volume fraction on friction welding properties of A360Al/SiC composites. The results show the design with lead angle-plane surface was achieved better joint strength compared to the design with plane-plane surface contact. Meanwhile, Ellis et al. (1994) indicated that the joint strength of 2648Al/14 % SiC particulate composites is about  $380 \text{ N/mm}^2$  with friction welding.

Since there are only very limited information has been reported on the application of the intermediate layer to friction welding, therefore Nishimoto et al. (2000) investigated the friction welding of silicon carbide to oxygen-free copper with an intermediate layer of reactive metals. They found that when a thin foil of reactive metals i.e. Al, Ti, Zr, or Nb, respectively, was applied as the intermediate layer, the bond strength of SiC to Cu was improved considerably. In contrast, when an intermediate layer of non reactive metals such as Fe, Ni, or Ag, respectively, was applied, the SiC specimen separated from the Cu specimen immediately after the bonding operation without the application of external load.

## 1.2 Problem Statement

Joining dissimilar materials has been long investigated through the combination of glass/metal, glass-ceramic/metal or ceramic/metal. Most of the time, joining different materials is not an easy task due to the different classes of atoms, ions or molecules in materials. When joining this dissimilar materials, it implies in property mismatches and structure discontinuities, which must be accounted for and minimized it.

Nowadays, there are several joining techniques of ceramics and metals have been developed. The most widely used method for joining ceramics to metals are brazing and diffusion bonding [Essa and Bahrani, 1991; Weiss and Sassani, 1998, and Mei and Xiao, 1999]. Brazing the oxide ceramics depends on the wetting and reactions with solid ceramic surfaces by liquid filler metal to produce bonds. A major problem of brazing an oxide ceramic with metal is the

resistance to wetting caused by the oxides on the surface of the ceramic [Chakravarty and Gupta, 2003].

Conventional joining methods either brazing or typically diffusion bonding require joining temperatures that exceed the ultimate use temperature of the joining assembly. If the intended use temperatures are high, the required joining temperatures are thus even higher. At these elevated joining temperatures, many metals will react with ceramics to form reaction layers that are often brittle [Locatelli et al., 1997]. It has been pointed out in several papers that in ceramics to metals joining by brazing and diffusion bonding, an excessive thickness of the reaction layer has a detrimental effect on the joint strength [Nishimoto et al., 2000].

Since brazing and diffusion bonding processes facilitate with heating cycle, therefore when longer heating cycle ( $> 30$  min) are encounter, relatively brittle joints may be formed [Roulin et al. 1999]. Shen et al. (2004) indicated that the oxide film can newly form during heating periods. A slight increase in the contact angle is generally observed during the subsequent heating or dwelling period. Indeed, the increase in contact angle would produce less wetting on the ceramic surfaces and finally produced the lower joint strength.

Active metal brazing requires a stringent firing atmosphere, either high vacuum or reducing gas conditions to prevent the active species. This represents a high capital expense and higher operating cost. In addition, recent studies on the oxidation behavior of active metal braze have shown that they

are unreliable at temperature beyond 500 °C, at which point they eventually oxidized completely, conferring little or no strength to the joint [Jin et al., 2004; Kim and Yoo, 1997].

In order to overcome those problems mentioned above, friction welding can be regarded as the ultimate technique for producing high integrity joints. It could be an interesting and cost effective alternative, provided that the strength of friction welded joints reaches or exceeds the strength of those joints produced by other techniques [Weiss and Sassani, 1998].

Deformation of the specimen is largely restricted to the volume of material adjacent to the original interface by an adiabatic shear process [Spindler, 1994]. Friction welding is used because of energy is produced in the specimen after opposite friction, resulting in less input energy, the heat affected zone (HAZ) is more narrow and hard, brittle intermetallic compounds are not easily produced, thus increasing the welding strength [Lin et al., 1999]. Friction welding has been proven practical to eliminate the formation of the intermetallic phases and to form a sound weld [Sundaresan and Murti, 1993; Fukumoto et al., 1997; Fukumoto et al., 1998].

Some of the advantages of friction welding are high in material saving, low production time and being possible of welding of different metals or alloys [Sahin and Akata, 2003]. Since friction welding can be achieved at high production rates, therefore it is economical in operation [Yilbas, 1995]. Protective atmosphere is unnecessary.

Common to all Al alloys is that an  $\text{Al}_2\text{O}_3$  layer on the Al weld component initially acts as a barrier to producing a bond. However, this layer is broken by the strong deformation occurring as a result of high rotational speeds and pressures, allowing for an oxide-free surface of the aluminum component to be welded [Yilmaz, 2003].

Fukumoto et al. (1997) indicated that materials such as aluminum or stainless steel have stable oxide films or contamination on the faying surfaces. The diffusion bonded in the solid condition should be held at a high temperature for a long time under uniaxial pressure to eliminate the stable oxide films. As a result, brittle intermetallic compounds are also formed and grow at the interface during the process. Meanwhile, friction welding is basically a solid state bonding process where the oxide films are eliminated within a few seconds owing to the rubbing together of the materials creating fresh contact surfaces.

Friction welding could avoid the formation of a liquid phase during the welding process and therefore can be carried out in air. The surfaces are joined in a plastic condition at hot forming temperatures. The typical defects caused by melting and solidification such as pores, pinholes, shrinkage cracks, segregation, grain coarsening and cast structure are therefore avoided and the risk of gas pickup is low due to the short welding cycle [Daymond and Bonner, 2003].

Therefore, in this study, the joining of metal to ceramic is between mild steel and alumina. The mild steel and alumina rod are selected for this study because of the cost of mild steel is cheaper compared to stainless steel, copper, titanium rods etc. Meanwhile, the alumina rod is prepared through slip casting is due to the price of commercial alumina rods are too expensive. The other reason for these selective materials is to produce base data system of the metal-ceramic joining through friction welding technique since there is still not be produce by other researchers.

### **1.3 Objectives of the Research**

Since friction welding widely used to join metal-metal in a wide range of applications, therefore the introducing of friction welding of mild steel-alumina give an alternative method of mild steel-alumina joining. There are not many researchers work in this field; therefore this study will focus on the performance of the mild steel-alumina joint including the strength of the joint, factors that determine to the successful joint etc. The objectives of this research are:

- (i) To study the optimum parameters i.e. friction times, interlayer thickness, rotational speeds at the constant friction and forging pressures to produce the maximum joint strength.
- (ii) To study the effect of different specimen diameters to the strength of the mild steel-alumina friction welded.

- (iii) To study the correlation between microstructural and interlayer thickness with the strength of the mild steel-alumina joint.

#### 1.4 Research Approach

This research was divided into two stages; firstly, the sample preparation and secondly is the joining process. In the first stage, the ceramics rods were prepared through slip casting and the mild steel rods were machined into required diameters. Figure 1.1 shows the flowchart of the whole process in this research.

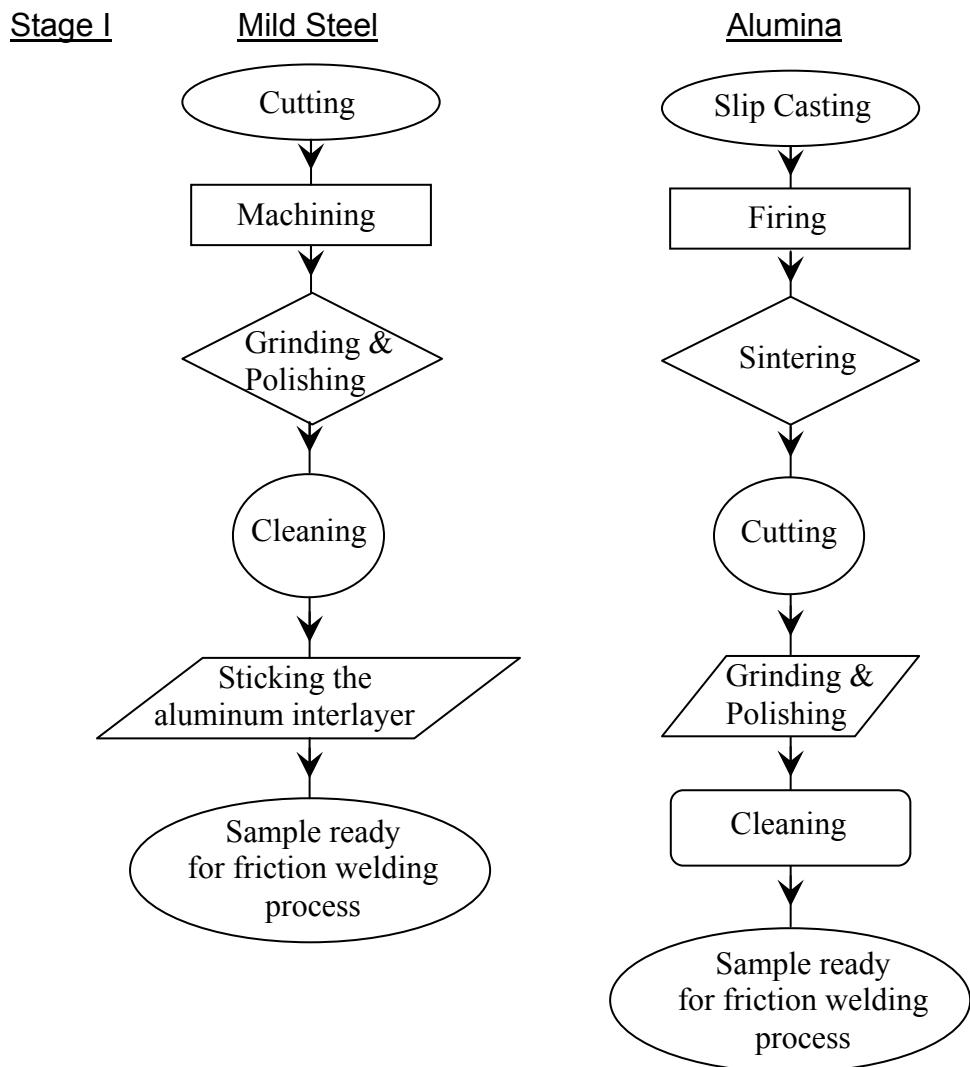


Figure 1.1: Flowchart of research stage I, raw materials preparation.

Stage II

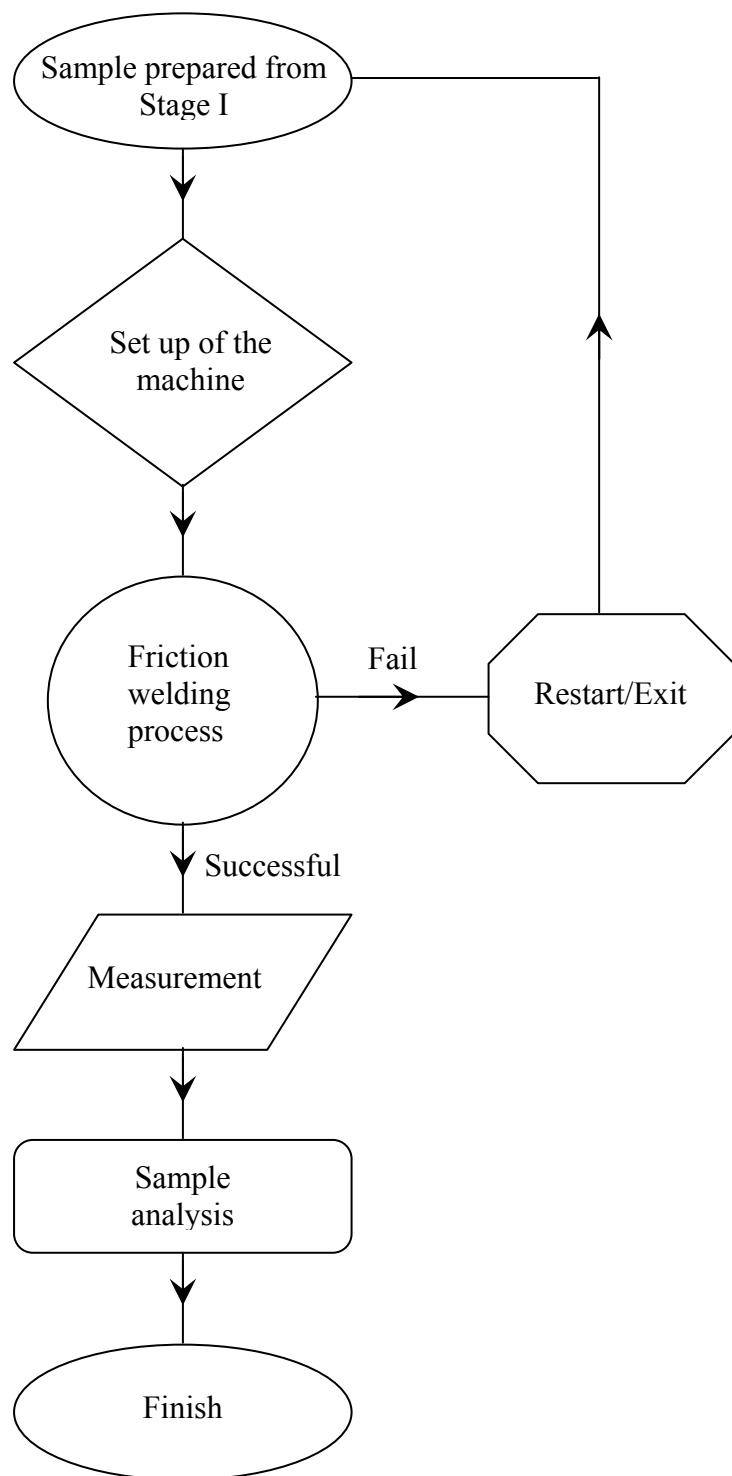


Figure 1.2: Flowchart of research stage II, friction welding process.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Introduction

Ceramic materials have many applications in engineering technology. History of ceramics art was begun in the Neolithic age about 5000 years BC in Asia Minor, about 4000 years BC in Egypt and about 3000 years BC in Europe. The improvement of technologies in the development of ceramics industry has upgraded the ceramic products into commercial sector. The distinguishes applications between traditional ceramics such as tableware, pottery, sanitary ware, tiles or bricks and advanced ceramics where depending on the properties for the specific application such as high strength at high temperatures, wear resistance, corrosion resistance etc. Indeed the advanced ceramic had offered excellent physical and mechanical properties for structural applications.

The major drawback of ceramics is the brittleness, i.e. failure without preceding plastic deformation. Another disadvantage is the large scatter in strength, which is caused by the brittleness and the scatter in the defect size. Both properties have to be considered in the design of ceramic components. In comparison with metals which are well known design criteria with wide range applications in industry. Therefore, further specific aspects have to be taken into account when selecting ceramic materials for a specific application and designing ceramic components.

In many technological branches, the demands to the materials used are ever rising. Therefore, one single material type is often not enough to meet

these requirements and, therefore, combinations of materials with different properties are required [Kohnle et al., 2002]. One interesting combination consists of metal and ceramic in order to combine ductility, high electrical and thermal conductivity with high strength and chemical inertness. This joining of materials is an important process commercially and technologically [Lee et al., 2001]. However, significant differences in chemical and physical properties between them make it extremely difficult to find an effective joining process that maintains the strength and resilience of the joint [Kim et al., 2003]. To overcome these difficulties, there are several joining techniques that have been developed to join ceramics for structural applications. Finally, having different materials at different regions of the same component is desirable for several applications. Joining to form these components is often the only way for fabrication. Metal/ceramic joints are used for example in automotive industry, cutting tools, turbine blades or dental implants.

## 2.2 Metal-Ceramic Joining

Metal-ceramic joining has been the subject of much development research over the years. In principle many techniques could be used to join metals to ceramics, ranging from welding to mechanical attachment. New joining methods and newer approaches to conventional methods have been developed over the years aiming at improved reliability. The availability of reliable metal-ceramic joining processes and its effect on the expansion of the structural ceramics market to large scale use in a quite establish concept [Paulasto and Kivilahti, 1998; Peteves et al., 1998]. Broadening the use of joining technology to new devices as well as improving joining to specific

applications has always been the major goal [Chuang et al., 1990; Rijnders and Peteves, 1999].

Engineering ceramics have been known over the past decades for having superior properties in comparison with metals namely, refractoriness, high wear resistance, lower density, low electrical and thermal conductivities, superior resistance to corrosion, high elastic modulus, and low coefficient of thermal expansion. Ceramics are ionically or covalently bonded inorganic and nonmetallic compounds of carbon, oxygen, nitrogen, boron and silicon. Because of the ionic and covalent bonding, they have relatively excellent properties as mentioned earlier [Mangonon, 1998]. Ceramic technologies have been widely used for aircraft and aerospace applications, wear-resistant parts, bioceramics, cutting tools, advanced optics, superconductivity, nuclear reactor, etc [Rosso, 2006]. The superior materials, however do suffer from drawbacks such as poor toughness and high expense, which are at present limiting their industrial applications. Meanwhile, metals in general are strong, ductile, and are good thermal and electrical conductivity, but they are typically not well suited for high temperatures, are prone to corrosion and chemical attack, and shrink or expand significantly with temperature changes. Therefore, metals and ceramics each have their advantages and disadvantages.

In most engineering fields, the application of ceramics is often restricted by the availability of an adequate joining technique. Most of the time, joining different materials is not an easy task. Atoms, ions or molecules in materials of different classes (ceramic, metal or polymer) are joined together in different

ways and therefore characterized by particular combinations of physical-chemical and mechanical properties [Nascimento et al., 2003]. The vast majority of joining processes involves heating of the couple. Upon cooling of the joint, mismatches in elastic modulus and thermal expansion coefficient often result in the development of residual stresses and retarded the mechanical strength of the joint. The key to a successful joint with dissimilar materials is the design of buffer interfaces capable of accommodating materials dissimilarities originated from different chemical bonds and properties [Paiva and Barbosa, 2000; Suganuma, 1993].

The joining of metal to metal has been established for a long time using various techniques, such as welding and brazing. Recently, new methods of ceramic–metal joining are being studied because of the increasing use of ceramics. There are several joining techniques of ceramics and metals such as mechanical joining, adhesive joining, friction welding, high energy beam welding, microwave joining, ultrasonic welding, explosive welding, reaction joining, combustion reaction joining, field assisted bonding, brazing, diffusion bonding, transient liquid phase bonding (TLPB), partial transient liquid phase bonding (PTLPB), etc [Zhang et al., 2006].

Many methods have been developed for ceramics to metals joints as mentioned above and each of these is useful for certain applications. As a correspondence, the application environments of materials or instruments become much more complex and critical. Many materials have to be applied in high temperatures, high pressures, high speeds or high erosive situations. So

the demand for improving the joining performances becomes more and more urgent since the quality of joining has a crucial influence on the application of materials or instruments in safety in the above situations [Jigang et al., 2006]. Reliable joining technologies are essential for full exploitation of the properties of the ceramics and for the success of the overall structure [Park et al., 2002].

### **2.2.1 Mechanical Fastening**

Mechanical joining methods are often based on localized, point-attachment process, in which the join is provided by a nail, a rivet, a screw, a clamp, a bolt or fastener. All such joints depend on residual tensile stress in the attachment to hold the components in compression. Many mechanical joints are designed for ease of assembly and disassembly such as bolts joints. Typical mechanical strength of the joints varies from 10 to 50 MPa [Nascimento et al., 2003].

Mechanical joining and adhesives with organics and cements have been widely used because of their ease and inexpensiveness. Mechanical joining has a variety of processes. For example, bolting and clamping are the simplest methods. The shrunk-in inserts was applied for the part of production of the turbocharger-rotor. The rocker arm chip made of silicon nitride was inserted in an aluminum die-cast arm. Thus mechanical joints can have heat resistance up to about 773 K. For the adoption of these methods, it is important to note that the mechanical joining sometimes accompanies a severe stress concentration and that ceramics is weak especially for tensile stress. Although the conventional adhesives can produce low strength below 20MPa generally, the

organic adhesive which has adhesive tensile strength of about 80 MPa became commercially available recently.

However, mechanical joint is not a generally attractive process for joining structural ceramics because of the frequent necessity for introducing intrusive stress-raising features such as threads or bolt holes, and the low strengths and lack of hermeticity of the joints [Nicholas, 1990].

Characteristic features of mechanical fastening include [Giles and Davis, 2004]:

- A heating cycle is generally not applied to the components being joined.
- This method usually requires special mechanical preparation, such as drilling hole, machining screw threads or chamfering abutting surfaces.
- The choice of suitable joint configurations is highly dependent on service conditions.
- The reliance on local stressing to effect joining requires thickening or some other means of reinforcement of the components in the joint region. This place is a severe restriction on the joint geometries that may be used and imposes a weight penalty on the assembly.

### **2.2.2 Brazing**

Brazing is a joining process that has proven to be one of the most versatile methods to join different kinds of metals or ceramics materials together. It is used in a variety of industries as diverse as aerospace and automotive. The successful performance of such systems depends on the

quality and reliability of the ceramic-to-metal joints [ElSawy and Fahmy, 1998].

Brazing is define as a process intended to permanently join two or more metals or materials together to form a single assembly by heating them in the presence of a filler metal that begins to melt above 450 °C. The liquid filler metal is then drawn into the gap between the closely fitted faying surfaces of the joint by capillary action. The base or parent materials (ceramics or metals) are not melted. Dissimilar metals that cannot be joined by a traditional welding process, because of their metallurgical incompatibilities or complex geometry, can be brazed successfully.

Two basic mechanisms are involved in bond formation, by wetting the surfaces with the braze alloy, and consequent interface reaction with the two surface layers of some micrometer thickness. If these layers increase in thickness the bond strength of the joints may be degraded, promoting failure by interfacial stresses due to thermal expansion mismatch. Brazing is well known in metallic bonding but is also one of the most promising methods of ceramic-metal joining because of its relatively non-stringent joint tolerance requirements and because ductile brazes are able to accommodate the thermal expansion mismatch occurring in a dissimilar joining system. The poor wettability of ceramic may be improved by the use of alloys containing an element capable of modifying the chemistry of the ceramic surface (active braze). Generally, Ag-Cu eutectic alloys containing small percentages of active metal (Ti, V, Nb, Ta), are used as braze materials [Lee et al., 1995].

The foremost challenge of making metal-to-ceramic joints rests with accommodating the thermal expansion mismatch between metal substrate materials and the engineered ceramics, and minimizing the resulting residual stresses [Vianco et al., 2002]. For joining ceramics to metals by brazing, the wettability and reactions of filler to ceramics is a key factor. In order to improve the wettability, the used of mixed active elements into filler which are Ni, Al, Ti, Hf, Th, V, Nb, Ta, Cr etc. These active elements can react with ceramics to form a reaction layer and then achieve strong chemical bonding between ceramics and metals [Loehman and Tomsia, 1988]. In stead of using active elements, the content of them must be reasonable, unless the increased of brittleness of the joint occurred. For example, the content of titanium in Ag-Cu-Ti filler is in the range of 1.5 % - 5.0 % [Zhang et al., 2006]. Since the filler Ag-Cu-Ti is used to join ceramics and metals successfully, the joint has less defects and higher joining strength at low temperature, where a maximum service temperature of only 400 – 500 °C.

Brazing with filler metals is an attractive process for joining structural ceramics for many applications. Most ceramics have problem in wetting conventional brazing filler metals. The other technique to solve problem was either by coating [Włosiński et al., 2000] the ceramic surface with a suitable metal layer prior to brazing (indirect brazing) or through the use of specially formulated filler metals that wet and adhere directly to an untreated ceramic surface (direct brazing). For indirect brazing, the ceramic is first coated in the joint area with a material that can be wetted by filler metal that does not wet the untreated surface. Coating techniques include sputtering, vapor plating, and

thermal decomposition of a metal-containing compound such as TiH<sub>4</sub> [Weiss and Adams, 1967; Ramsey and Lewis, 1985]. Alumina ceramics can be joined with this technique with molybdenum-manganese (Mo-Mn) coating [Moorhead and Kim, 1991].

Direct brazing requires the enhancement of both wetting and adherence of active metals to ceramics without the need for coating the ceramic surface. Whether the ceramic is an oxide, carbide or nitride, the active metal reacts with the ceramic surface, forming an interfacial layer that can be wetted by the bulk of the filler metal. Titanium is the widely used active element addition to filler metals formulated to braze directly high melting oxide ceramics [Nicholas, 1986; Lee and Yu, 1995; Brochu et al., 2004]. The critical interfacial reaction product in the case of oxide ceramics brazed with Ti-containing filler metals is either TiO or Ti<sub>2</sub>O<sub>3</sub> with appreciably higher adhesion in systems that result in the formation of TiO [Nicholas, 1986]. Moreover, mullite and ZrO<sub>2</sub>-toughened mullite (ZTM) were joined with Ag-Cu eutectic braze alloys that contain Ti or Zr as active elements with the resulting four-point bend strength of 108 MPa [Loehman, 1994].

Not only has active metal been used as the filler materials for brazing, but brazing with nonmetallic glasses as filler materials has been studied with structural ceramics, such as alumina, Partially Stabilized Zirconia (PSZ), Si<sub>3</sub>N<sub>4</sub>, and sialon [Zdaniewski, 1987; Milberg, 1987; Baik and Raj, 1987]. The rationale for this approach is that many ceramics have amorphous phases remaining at the grain boundaries after the sintering process, so they would be

expected to be wet by, and compatible with, similar glassy materials, introduced at the joint. However, this joining technique has a problem where some of the glasses, even those formulated to match the grain-boundary composition of a specific ceramic, have coefficients of thermal expansion significantly different from those of the ceramics, which can cause high residual stresses and cracking in the joint. The most extensive studies on brazing with glasses have been those on  $\text{Si}_3\text{N}_4$ . The glasses include those similar to that found at the grain boundaries of a hot pressed  $\text{Si}_3\text{N}_4$  [Johnson and Rowcliffe, 1985].

In brazing technique, there are a few factors that must take into consideration in choosing the fabrication conditions to be employed to braze a ceramic component. There are three the most important criterions: surface preparation, environment and thermal cycle [Nicholas, 1990].

### **2.2.2.1      Surface Preparation**

Surface preparation methods for metal components prior to their brazing to ceramics are well established. There is clear evidence that surface roughness affects wetting and flow behavior during brazing. The roughness of metal surface can be varied by machining or abrasive operation such as milling, grinding and polishing. Chemical treatments are used to ensure that metal surfaces are not coated by thick oxide films. Meanwhile, the thin films will reform virtually instantaneously. The surfaces are then treated with degreasing agents to remove organic contaminants that could degrade to form carbonaceous layer during brazing.

The roughness of the ceramic surfaces can also affect wetting and flow behavior. Normally the rougher surfaces will degrade wettability. Ceramic component surfaces generally have to be ground to produce a surface roughness but sometimes can also nucleate cracks which degrade mechanical properties [Kay, 2003].

### **2.2.2.2 Environment**

The environment in which active metal brazing is conducted is of considerable important. In most operations a vacuum is employed to achieve low oxygen and nitrogen activities, with furnace pressures of less than  $10^{-3}$  mbar being recommended and vacuum of  $10^{-4} - 10^{-5}$  mbar being employed when possible [Jadoon et al., 2004]. Therefore, in determining the conditions to be used and then controlling them, it is useful to monitor not only the vacuum level but also the partial pressures of the residual gasses.

### **2.2.2.3 Thermal Cycle**

The thermal cycle can be closely controlled and hence its selection is particularly important. The specific heating rates employed will depend upon the capabilities of the equipment and the size and geometry of the specimen. In practice, the using of resistance furnace which heating and cooling rates may be restricted by the need to avoid thermal shock to large components [Nicholas, 1990].

A common and often successful heating process is moderately slowly with  $10 - 20 \text{ Kmin}^{-1}$ , to a temperature just below the braze solidus and to hold

for at least 15 minutes to ensure uniformity of temperature within the specimen [Wan Jaafar et al., 2002]. This subsolidus hold is then followed by even slower heating to the brazing temperature, which is often 323° K or more above the liquidus, and the hold for about 30 minutes. The slow heating rates which about 1 – 5 Kmin<sup>-1</sup> helps to maintain temperature uniformity throughout the component and to avoid premature flow of the more readily melted [Jadoon et al., 2004]. The long dwell time at the brazing temperatures relates to the usually poor liquidity of brazes or the need for them to react with the specimen.

### **2.2.3 Diffusion Bonding**

Diffusion bonding of metal has a long history. It was one of the first joining technologies developed by man and examples of artifacts fabricated in this manner have been discovered in very early sites of metal working [Derby, 1990]. Diffusion bonding is a joining process in which two nominally flat surfaces are held together at an elevated temperature (typically above 60% of the melting point of the least refractory materials) for a period of time until a bond is formed. It is a solid-state joining process that capable of joining a wide range of metal and ceramic combinations to produce both small and large components. This joining technique needs generally the use of a metallic interlayer, which can interact with the ceramic part during the bonding process [Raevska, 1998]. The properties of the joint at high temperature depend on many factors, such as the thermal expansion mismatch between ceramic and interlayer material, or the reactivity of both materials at high temperature [Beaume et al., 2003].

In its simplest form, diffusion bonding involves holding pre-machined components under load at an elevated temperature usually in a protective atmosphere or vacuum. The loads used are usually below those which would cause macrodeformation of the parent materials and temperatures of 0.5 - 0.8  $T_m$  (where  $T_m$  = melting point in Kelvin) are employed. Times at temperature can range from 1 to 60 minutes, but this depends upon the materials being bonded, the joint properties required and the remaining bonding parameters. Although the majority of bonding operations are performed in vacuum or an inert gas atmosphere, certain bonds can be produced in air. The bonding temperature influences yield strength and atomic diffusion of the alloys; therefore, it influenced the homogeneity of the composition and microstructure of the joint [He et al., 2002].

Bonding in the solid phase is mainly carried out in vacuum or a protective atmosphere, with heat being applied by radiant, induction, direct or indirect resistance heating. Pressure can be applied uniaxially or isostatically. In the former case, a low pressure (3 – 10 MPa) is used to prevent macrodeformation of the parts (i.e. no more than a few percent). This form of the process therefore requires a good surface finish on the mating surfaces as the contribution to bonding provided by plastic yielding is restricted. Typically surface finishes of better than 0.4  $\mu\text{m}$  are recommended and in addition the surfaces should be as clean as practical to minimize surface contamination.

In hot isostatic pressing, much higher pressures are possible (100 – 200 MPa) and therefore surface finishes are not so critical, finishes of 0.8  $\mu\text{m}$  and

greater can be used. A further advantage of this process is that the use of uniform gas pressurization allows complex geometries to be bonded, as against the generally simple butt or lap joints possible with uniaxial pressurization.

Since the diffusion bonding conducted at high temperatures and under high pressures [Peteves, 1996], the components fabricated are typically limited to simple shapes. Reaction bonding, also a high temperature joining process, often yields joints that contain residual porosity, unconverted reactants and undesired secondary product phases, any of which can reduce joint strength by acting as sites for crack initiation [Lewinsohn et al., 2000; Maeda et al., 2003]. Joints formed by converting a polymeric precursor to the final ceramic bonding phase often experience cracking during processing because of the large volumetric shrinkage that accompanies pyrolysis [Jin et al., 2004].

Diffusion bonding is an important fabrication technique for making components in electronic, nuclear and aerospace industries. This technique provides novel joining operation for similar and dissimilar materials without gross microscopic distortion and with close dimensional tolerance [He and Liu, 2006]. The knowledge of diffusion behavior is of interest for production of these materials and for their practical use [Nakajima et al., 1996]. This method has already proved considerable potential for joining of Ti and Al base alloys [Salehi et al., 1992; Wang et al., 1996; Huang et al., 1998]. Joining of varieties of dissimilar and similar materials has already been carried out by diffusion bonding, though many of the parameters of the thermomechanical processing

are yet to be established on commercial scale [Tortorici and Dayananda, 1998; Calvo et al., 1988; Nakao et al., 1991; Eckman et al., 1992].

Diffusion bonding is considered as an advantageous technique for producing that type of joint since high-temperature mechanical resistance and defect-free interfaces can be achieved. The main drawback of this technique is the high magnitude of thermal stresses across the interface, typical of this kind of joints, resulting from the high temperatures used to produce the joint and the difference in thermal expansion coefficients of the components [Breme et al., 1998; Abed et al., 2001; Park et al., 2002]. However, when compared to other joint technologies, as active metal brazing, diffusion bonding does not involve the introduction of foreign chemical elements in the interface, which is built by the reaction/diffusion/dissolution of the elements constituting the components to be joined. This can be advantageous from the point of view of corrosion resistance of the joint because no unfamiliar chemical elements are brought to the system. It should be stressed that when bearing in mind biomedical applications, practical applications of such metal/ceramic interfaces may be found in a wide variety of devices ranging from external or implantable sensors to dental implants. For these applications, corrosion resistance becomes a particularly important characteristic to be considered [Rocha et al., 2003].

In general, metal/ceramic interfaces presents different layers, which have very small dimensions and complex variations in chemical compositions between them. As a consequence, the electrochemical characterization of the whole interface becomes very difficult, as well as the understanding of the