# TO STUDY THE EFFECT OF HYDROGEN CHARGING ON MECHANICAL BEHAVIOR OF AA6061 ALLOY AND AA6061-SS304 WELD JOINT

**M.Tech Thesis** 

By ABHISHEK KUMAR SAHU



DEPARTMENT OF METALLURGY ENGINEERING AND MATERIALS SCIENCE

# INDIAN INSTITUTE OF TECHNOLOGY INDORE

**JUNE 2022** 

# TO STUDY THE EFFECT OF HYDROGEN CHARGING ON MECHANICAL BEHAVIOR OF AA6061 ALLOY AND AA6061-SS304 WELD JOINT

## A THESIS

Submitted in partial fulfillment of the requirements for the award of the degree

*of* Master of Technology

by ABHISHEK KUMAR SAHU



# DEPARTMENT OF METALLURGY ENGINEERING AND MATERIALS SCIENCE

# INDIAN INSTITUTE OF TECHNOLOGY INDORE

**JUNE 2022** 



# **INDIAN INSTITUTE OF TECHNOLOGY INDORE**

#### **CANDIDATE'S DECLARATION**

I hereby certify that the work which is being presented in the thesis entitled **TO STUDY THE EFFECT OF HYDROGEN CHARGING ON MECHANICAL BEHAVIOR OF AA6061 ALLOY AND AA6061-SS304 WELD JOINT** in the partial fulfillment of the requirements for the award of the degree of **MASTER OF TECHNOLOGY** in **METALLURGY ENGINEERING** and submitted in the **DEPARTMENT OF METALLURGY ENGINEERING AND MATERIALS SCIENCE, Indian Institute of Technology Indore**, is an authentic record of my own work carried out during the time period from July 2021 to May 2022 under the supervision of Dr. Jayaprakash Murugesan, Assistant Professor, Department of Metallurgy Engineering and Materials Science, Indian Institute of Technology, Indore.

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other institute.

Abhishek Sahu 27/06/2022 Signature of the student with date (ABHISHEK KUMAR SAHU)

This is to certify that the above statement made by the candidate is correct to the best of my/our knowledge.

M. Jayapraken 27.6.222

Signature of the Supervisor of M.Tech Thesis (with date) (Dr. Jayaprakash Murugesan)

ABHISHEK KUMAR SAHU has successfully given his M.Tech. Oral Examination held on 8<sup>th</sup> July, 2022.

M. Jappenhall

(Dr. Jayaprakash Murugesan) Signature of Supervisor of M.tech. Thesis Date: 27.6.2022

Signature of PSPC Member Date: 27.6.2022 (Dr. Indarasen Singh)

S Samal Convener, DPGC Date: 12 07 2022

Abuijit Cu

Signature of PSPC Member Date: 27.06. 2022 (Dr. Abhijit Ghosh)

1

v

## ACKNOWLEDGEMENTS

My very special gratitude to my project guide Dr. Jayaprakash Murugesan, Assistant Professor, Department of Metallurgy Engineering and Materials Science, Indian Institute of Technology, Indore, for his support, suggestions, discussions and for his valuable guidance in the laboratory. His guidance, support, encouragement and valuable advices throughout this project in all aspects of the work helped me immensely. I am also grateful for his kindness and patience during these months. His time management skills always amazed me. His knowledge and the skills to communicate them simply and effectively are great and really helped me to gain a conceptual understanding of many topics needed to carry out my research effectively.

I am deeply grateful to thank my PSPC members, Dr. Abhijit Ghosh and Dr. Indrasen Singh for their valuable time in assessing my work and for the valuable suggestions and insightful comments. Their friendly interactions and constructive support have enabled this work to achieve its present form

I am thankful to all faculty members and staff of the Department of Metallurgy Engineering and Materials Science for their guidance, help and support. I am thankful to MHRD, Govt. of India, for providing fellowship during my Master studies.

Last but not least, I cannot forget the support and encouragement received from my family and my colleagues.

#### Abstract

Aluminium alloy is one of the most extensively used engineering materials. It has high degree of machinability when it is compared to other lightweight materials like magnesium and titanium. Zinc, silicon, magnesium, iron, copper, tin, and manganese are frequent alloying elements. These extra elements improve the workability, electrical conductivity, corrosion resistance, and strength of aluminium. As a result hybrid structure is being preferred and replacement with lighter materials is being done. Aluminium exhibits the suitable candidate for this selection. But there is a problem of joining of Aluminium with the materials which involves numerous techniques such as riveting, nut and bolt, welding etc. In welding also there are numerous techniques as fusion welding and solid-state welding which is further classified into various other types.

Now-a-days hydrogen is seen as a future fuel in vehicle. So it becomes important to store it safely since it causes hydrogen embrittlement and affects the properties of the material significantly. To overcome this, the objective is to analyse the mechanical properties of AA6061 alloy and the welding technique which was preferred for joining AA6061-SS304 was Friction Stir Welding which is a solid state welding.

AA6061 alloy and AA6061-SS304 weld joint was electrochemically charged with hydrogen for various time intervals and then their microstructure, tensile test, hardness and XRD was performed to analyse the mechanical properties. By performing the above test it was found that with the increase in charging time the mechanical properties such as tensile strength and hardness was increasing but the % elongation was decreasing which indicates the brittleness nature of the material. XRD test result also suggested that for 140 hours for AA6061 there is presence of hydrogen in the form of Aluminium hydride for which the tensile strength

obtained was 389.232 MPa and when compared to that of the base metal it was found to be 333.524 MPa.

## TABLE OF CONTENTS

LIST OF FIGURES	xiii
LIST OF TABLES	XV
ACRONYMS	xvii

Chapter 1: Introduction	19
1.1 Aluminium and its Alloys	19
1.2 Steel	23
1.3 FSW	24
1.4 Hydrogen Embrittlement	27
1.5 Organization of Thesis	27

#### **Chapter 2: Review of Past Work and Problem Formulation.29**

2.1: Review of Past Work	29

2.,1,2: Problem	Formulation	34

Chapter 3: Research Methodology......35

Chapter 4: Research Objective37
---------------------------------

Chapter 5: Experimental Details	9
5.1 Material Preparation3	9
5.2 Microstructure and Characterizations	9
5.3 Welding4	0
5.4 Mechanical Properties4	0

Chapter 6: Results and Discussion41	
6.1 Specimen Preparation41	
6.2 Microstructural Analysis46	
6.3 Tensile Test48	
6.4 SEM51	
6.5 .Hardness54	
6.6 XRD57	

Chapter 7: : Conclusions and Scope for Future Work	59
7.1 Conclusions	59
7.2 Scope for Future Work	59

REFERENCES	61
------------	----

#### LIST OF FIGURES

Figure 1.1: Friction stir welding fixture at IITI central workshop

Figure1.2: Friction stir welding process

**Figure 2.1:** Effect of hydrogen charging on tensile propertie of 99.99% aluminium. (a) Ultimate tensile stress, (b) 2% yield stress, and (c) failure strain. The error bars represent one standard deviation

**Figure 2.2:** Depth of hardening after aging at 200°C, between the first and second stages of recovery

Figure 6.1: Eletrochemical Charging Setup

Figure 6.2: Hydrogen charged specimen for tensile test

**Figure 6.3:** Hydrogen charged specimens for Microstructure, Hardness and XRD analysis

Figure 6.4: Friction Stir Welding Machine

Figure 6.5: Setup showing plates about to be welded

Figure 6.6: Welded plates

Figure 6.7: Tensile test specimen of AA6061-SS304 weld joint

**Figure 6..8:** Dimensions of tensile test specimen of AA6061-SS304 weld joint

**Figure 6.9:** Microstructure of charged specimens at 20x of (a) Base (b) 40h (c) 80h (d) 140h

Figure 6.10: Graph showing the variation of tensile strength and

strain % of hydrogen charged specimens for different charging time intervals for AA6061

**Figure 6.11:** Stress-strain curve of hydrogen charged specimens for different charging time intervals for AA6061-SS304 weld joint

**Figure 6.12:** Fractured Surfaces observed under SEM of (a) Base (b) 40h (c) 80h (d) 140h

**Figure 6.13:** Vickers Hardness vs Depth curve of hydrogen charged specimens for different charging time intervals for AA6061 alloy

**Figure 6.14:** Vickers Hardness vs Depth curve of welded charged specimens for different charging time intervals.

Figure 6.15: XRD plot of AA6061 alloy

# **LIST OF TABLES**

**Table 1.1:** Wrought alloys of Aluminium series and their applications.

**Table 6.1:** Variation of strength and elongation % with respect to chargingtime for AA6061 alloys.

**Table 6.2:** Variation of strength and elongation % with respect to chargingtime for AA6061-SS304 weld joint.

**Table 6.3:** Variation of Hardness of AA6061 with charging time.

**Table 6.4:** Variation of Hardness of AA6061-SS304 weld joint with charging time

# ACRONYMS

- 1. FSW: Friction Stir Welding
- 2. MIG: Metal Inert Gas
- 3. SEM: Scanning Electron Microscope
- 4. XRD: X-Ray Diffraction

### **Chapter 1**

## Introduction

#### **1.1** Aluminium and its Alloys

Aluminium has lower specific weight and corrosion resistance than steel. Aluminium alloy is one of the most extensively used engineering materials. Furthermore, they are less difficult to machine. Aluminium alloys have been discovered to provide the best performance. It has high degree of machinability when it is compared to other lightweight materials like magnesium and titanium. Zinc, silicon, magnesium, iron, copper, tin, and manganese are frequent alloying elements. These extra elements improve the workability, electrical conductivity, corrosion resistance, and strength of aluminium. Wrought alloys and casting alloys are the two primary categories of aluminium alloys [1]. Heat-treatable and strain hardenable alloys are split into both of these categories of aluminium alloys.

Aluminium alloys have long been used to make engineering components and structures where corrosion resistance and light weight are important. They've shown to be extremely useful in the production of aeroplane components [2].

A four-digit number is assigned to each alloy. The first digit represents the major alloying components; the second digit, if not zero, denotes an alloy variation; and the third and fourth digits represent the exact alloy in the series. Aluminium alloys are utilised in a variety of applications, including marine, automotive, aviation, gas and air cylinders, and many others.

- Marine Applications: Marine alloys are used to build boats and ships, as well as other saltwater-sensitive land and marine applications. Aluminum alloys 5052, 5059, 5083, 5086, 6061, and 6063 are among them. The aluminium alloys 6082, 6005A, 5183, and 4043 are also used in offshore and maritime structure [3].
- 2) <u>Automotive and Aircraft alloys:</u> Aluminium alloys like as 6111 are widely utilised in car exterior and body panels. Inner body panels are made of 5754 and 5083 aluminium alloys, while bonnets are made of 6111, 6016, and 2036 alloys. 5456 Al is extensively used in trailer and vehicle body panels. Automotive frames are made of 5754 and 5182 aluminium formed sheets, as well as 6063 or 6061 extrusions.
- 3) Gas and Cylinder applications: Aluminum 319 and 356 are commonly used for crankcases and cylinder blocks, with aluminium 242 being utilised on rare occasions. The high strength-weight ratio and ease of fabrication for replacement of steel and cast-iron parts is a significant benefit [4-6].

Table1.1:	Wrought	alloys	of	Aluminium	series	and	their
applications.							

Wrought alloys of	Elements alloyed	Applications
Aluminium Series	with	
1000 Series	Minimum of 99 % Al	They can be honed via
		hard work.
		Plate, foil, electrical
		conductors, and
		aviation components
		are among of the
		applications.
2000 Series	Copper	Precipitation hardening
		is an option. They've
		been used in aerospace,
		plates, bar and wire,
		and forgings.
3000 Series	Manganese	They can be made to
		work harder.
		Beverage cans, stiff foil
		containers, and
		decorative items are all
		examples of
		applications.
4000 Series	Silicon	Sheet, cladding, filter, a
		nd rod made of work-
		hardened forging.

5000 Series	Magnesium	Precipitation hardening is an option. Rivet, rod, rocket cryogenic tanks, welding, marine, sheet and vehicle component
6000 Series	Magnesium and	foil, rivets, rod. Automotive, sheets, ma
0000 Series	Silicon	<ul><li>Automotive, sheets, ma</li><li>rine, forgings, and rod a</li><li>re some of the applicati</li><li>ons.</li><li>Precipitation hardening</li></ul>
		is an option.
7000 Series	Zinc	Precipitation hardening is an option. Forging, thick plate, aerospace.
8000 Series	Lithium	High temperature electrical wire for aeronautical ap plications

The 6061 aluminium alloy has been utilised for general purposes o ver all other types of aluminium alloys because of the unique chara cteristics it possesses when compared to others. The 6061 aluminium alloy has been utilised for general purposes over all other types of aluminium alloys because of the unique characteristics it possesses when compared to others. The influence of machining conditions on the corrosion of 6061 aluminium alloy has revealed that electrical discharged machining provides greater resistance to pitting corrosion than diamond and carbide turning machines.

#### 1.2 Steel

Due to its appealing qualities, austenitic stainless steels (AISI 304) are the most adaptable rating in stainless steel and are widely employed in a variety of sectors. It possesses a high level of durability, ductility, toughness at cryogenic temperatures, and corrosion resistance. Furthermore, this steel offers great weldability as well as strong fatigue and oxidation resistance. Contrarily, due to its high work hardening, high built-up edge, and low heat conductivity, AISI 304 stainless steel is thought to be the hardest alloy steel to cut. Different cutting fluids are used to increase machining efficiency and surface integrity, lower cutting temperatures, lengthen the life of cutting tools, and reduce tool-toworkpiece friction. Contrarily, due to its high work hardening, high built-up edge, and low heat conductivity, AISI 304 stainless steel is thought to be the hardest alloy steel to cut. Different cutting fluids are used to increase machining efficiency and surface integrity, lower cutting temperatures, lengthen the life of cutting tools, and reduce tool-to-workpiece friction. As a result, the goal of this review paper is to compile the findings of numerous investigations on the machining capabilities of AISI-304 stainless steel carried out by different academics. Future researchers can select the appropriate machining parameters, coolant type, and machine to improve overall machined surface integrity by reviewing a variety of applications and aspects that affect the performance of AISI-304 stainless steel.

#### **1.3 FSW**

With the financial help of NASA, Wayne Thomas invented Friction stir welding (FSW), a relatively recent method to join materials in 1991. The goal of this procedure was originally concerned to orbital spacecraft for reducing its weight and it was limited to aluminium alloys, but as the domain of the friction stirs welding process grew, so did the tool materials. The materials used in this welding process are copper and its alloys, titani um and its alloys, magnesium and its alloys, lead, nickel alloys, zinc, stai nless steel, and mild steel.

Friction stir welding is a solid state joining technology that uses a combination of heat generated by friction and mechanical mixing to fuse two or more comparable or dissimilar materials with varying melting temperatures. This process is commonly used to weld hard materials such as steel. Because being light in weight these materials can be used in a variety of applications. Lightweight materials with good mechanical properties are frequently employed in transportation and other industries.

Preference is given to mechanical qualities. The mechanical properties of the welds are diminished when these materials are joined using fusion welding procedures. Friction stir welding does not alter the mechanical properties of the materials to be welded because only the forging action is used and no materials are melted.

Unlike traditional fusion welding, which is commonly used to fuse materials like structural steel, FSW is a unique welding procedure that employs superplastic severe deformation mixing and frictional heat is used to connect two materials. Aluminium and magnesium alloys are lightweight alloys 6500 C and 6400 C, respectively, are low melting points. Because of the low melting point of the material, Fusion welding generates joints with hot fissures, a poor surface quality, low shear corrosion, bending, and strength [7]. As a result of its reduced working

temperatures and great efficiency, FSW has emerged as a viable alternative for lightweight alloy joining. In friction stir butt welding technique, the spinning tool's motion against the workpiece plates is depicted in the diagram. The tool motion creates severe plastic deformation and frictional heat, which causes the workpiece plates to distort and mix, forming a junction.

The FSW technique has been discovered to be highly adaptable due to its ability to efficiently weld essential structural and lightweight alloys. This method has been used in a variety of settings with encouraging outcomes. The FSW joint's configuration is determined by the application requirements. Changes in workpiece plate positioning, tool dimensions, cooling method, and process parameters are all part of the weld configuration modification. Butt welding, lap welding, and spot welding are some of the most common weld designs.



Figure 1.1: Friction stir welding fixture at IITI central workshop

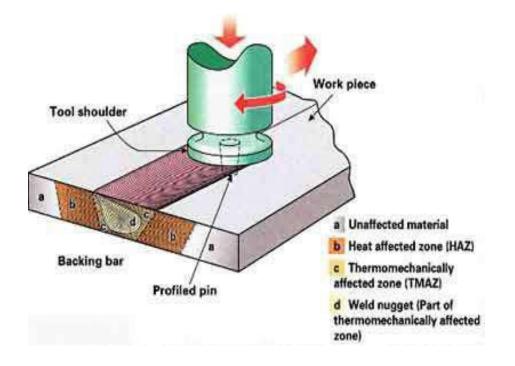


Figure 1.2: Friction stir welding process

Mechanism of Friction stir welding (FSW): It is a solid-state joining technique that uses a tool of non-consumable type to join two workpieces or facing plates without melting the materials. Friction between the revolving tool and the workpiece generates heat, resulting in a softened zone in the workpiece around the FSW tool. The tool mechanically intermixes the two separate pieces of metal as it moves, forging the heated and softened metal by applying mechanical pressure, similar to how clay or dough is bonded. Friction Stir Welding (FSW) of flight and non-flight gear is governed by this process definition, which specifies the minimal requirements. Pressure vessels and pressurised components are not included. The specification includes procedural and quality assurance requirements.For example,tool material is HSS, Grade M 35, outer should er diameter is 50 mm, inner shoulder length is 10 mm, inner shoulder dia meter is 12 mm, pin profile is tapered cylindrical, pin length is 4.75 mm,

pin diameter is 4 mm, and the ratio of outer shoulder diameter to pin dia meter is 3.

#### 1.4 Hydrogen Embrittlement

Metals become brittle as a result of the entry and diffusion of hydrogen into the material, which is known as embrittlement. The amount of hydrogen absorbed as well as the microstructure of the material impact the degree of embrittlement. Microstructures that provide high strength, as measured by hardness, or have precise distributions of grain boundary particles or inclusions, might increase embrittlement susceptibility.

When the phenomenon develops to cracking, it becomes significant. When enough stress is applied to a hydrogen-embrittled item, this happens. Residual tensions, accompanying fabrication activities such as forming and welding, and applied service stresses can all contribute to such stress levels. The effect of hydrogen on the mechanical properties also depends upon the temperature.

#### **1.5 Organization of Thesis**

Chapter 1: Introduction to AA6061, SS304 and FSW.

Chapter 2: Literature review of past works and problem formulation of this research work.

Chapter 3: Research methodology followed.

Chapter 4: Objective of research work.

Chapter 5: Includes procedures followed to perform the experimental work

Chapter 6: Includes results and discussions of the above obtained result.

Chapter 7: Includes conclusions and scope for the future work.

#### **Chapter 2**

# **Review of Past Work and Problem Formulation**

## 2.1 Review of Past Work

Over the time because of good mechanical qualities and low weight, aluminium alloys are increasingly being used as a structural material in metal matrix composite materials. Aluminum alloys have been extensively used. Aluminium alloys are susceptible to hydrogen damage and it becomes necessary to study it extensively.

Yizhe Chen, Shilong Zhao, Huijuan Ma, Hui Wang, Lin Hua and Shuang Fu studied the effect of hydrogen embrittlement on aluminium alloys for vehicle mounted storage tanks. They analyzed the mechanism which inclu des hydrogen enhanced local plasiticity model, hydrogen enhanced decoh esion mechanism and hydrogen pressure theory. The detection included sl ow strain rate test, linearly increasing stress test and so on while the meth ods of prevention included coating process.

The security of transportation equipment [8-10] has drawn increasing attention.

Hydrogen-powered cars are seen as one of the most promising solutions [11-14] to the energy dilemma. The hydrogen vehicle offers benefits over electric vehicles in terms of technical simplicity and high filling rates [15] while replenishing with energy. Hydogen Embrittlement severly affects the petrochemical, aerospace, automotive, hydrogen energy, and other industries' service safety of aluminium alloys [16].

It was found that hydrogen embrittlement is a severe type of failure affecting the aluminium alloys. It also represents a huge danger to the safety of vehicles and passengers too. The hydrogen enhanced local plasticity model (HELP) was widely accepted. This theory holds that through stress-induced diffusion, hydrogen atoms are concentrated to the crack tip, leading to fracture of materials. And for its detection linearly increasing stress test (LIST) was having advantages over the other processes being its ease of process and simple to process. Hydrogen embrittlement prevention methods mainly included techniques for preventing surface film formation, coating prevention, heat treatment, and grain refinement. Among them, the inhibitory approach using sodium silicate solution works well. Stops hydrogen during manufacture from entering small fissures of the tanks used to store hydrogen. Additionally, it is thought that additional preventative methods will be created via the fusion of many preventative measures.

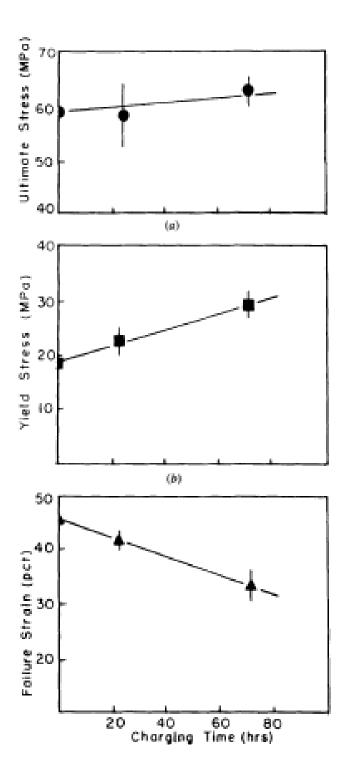
The characteristics of hydrogen in aluminium and its alloys, as well as its impact on mechanical properties, were explored by Rajan Ambat and E S Dwarakadasa. Examined is the relative significance of anodic dissolution versus hydrogen embrittlement in explaining the stress corrosion cracking mechanism of these alloys. It was found that mechanical qualities of aluminium and its alloys deteriorate in the presence of hydrogen. Additionally, reversible hydrogen embrittlement is considered to be a risk with these alloys.

J.W. Watson, Y.Z. Shen, and M. Meshii studied the tensile and microhardness tests to examine the effects of cathodically charged hydrogen on the mechanical characteristics of pure aluminium. In tensile experiments, it was discovered that hydrogen charging decreased ductility and increase.yield and tensile stresses.

30

Microhardness tests revealed that cathodic charging produced a severely h ardened surface region. The depth of this region was expanded by additio nal charging once the hardness in this area quickly saturated.

It was found that many modern aluminium alloys show suscepibility to hydrogen damage and it becomes job of extensive study [17-22] of immense importance. Pure aluminum's characteristics and microstructure have been demonstrated to be impacted by hydrogen. In terms of these impacts, a highly toughened surface region corresponding to an enhanced dislocation density is prevalent. The surface area moves inward from the charging surface at a rate similar to the hydrogen diffusion rate in aluminium. It was examined that hydrogen charging led to an increase in the yield stress, a slight increase in the ultimate tensile stress, and a decrease in the strain at failure. The respective graphs so obtained are shown below.



**Figure 2.1:** Effect of hydrogen charging on tensile propertie of 99.99% aluminium. (a) Ultimate tensile stress, (b) 2% yield stress, and (c) failure strain. The error bars represent one standard deviation

Furthermore microhardness test was also being done which showed the variation of microhadrness when aluminium alloys were subjected to various parameters such as when charged only for 48 hours, when charged for 48 hours and aged at 200°C and in annealed condition with depth.

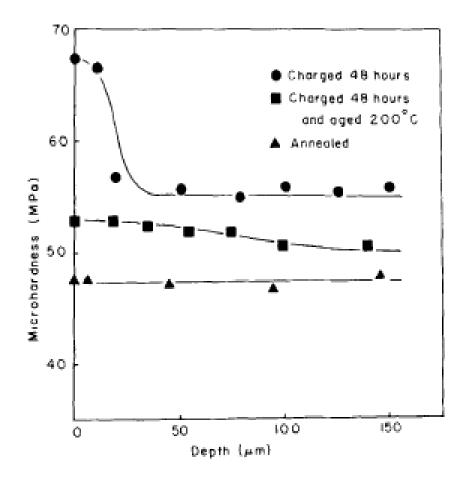


Figure 2.2: Depth of hardening after aging at 200°C, between the first and second stages of recovery

The parameter optimization of the preheating method for metal inert gas (MIG) dissimilar welding on aluminum-stainless steel was explored by Mohamad Rusdi Abdul Rahman, Luqman Hakim Ahmad Shah, and Mahadzir. This study examines the parameter optimization of a preheating technique on stainless steel SS304 before welding aluminium to stainless

steel that is not the same alloy. Metal inert gas (MIG) welding with a butt joint type weld was the technique used. the 111.27 MPa maximum ultimate tensile strength. Due to the near zero solubility and sharp difference in melting temperatures between aluminium and steel, combination was challenging. As a result, this makes it easier for the infamous FexAly brittle intermetallic (IMC) combination to form while welding the two metals together. [23], [24]. Several studies have suggested the use of preheating before the welding process to reduce the creation of such fragile structures. The preheating approach was used to the tungsten inert gas (TIG) dissimilar welding of AA6061 and SS304 by Shah et al. [25] and Razak [26], respectively. It was discovered that both results indicated an increase in the tensile strength of the joint. when preheating steel to 150 C. Other studies by Kreimeyer and Vollertsen [27] and Bang et al. [28] also proposed the use of a secondary heat source to pre-heat the steel counterpart before welding in order to increase the output of the joint strength.

### 2.2 **Problem Formulation**

These two materials SS304 and AA6061 have lot of potential for hydrogen storage and hydrogen transport. Hydrogen has adverse affect on many materials and hence it is very important to test the effect of hydrogen on these materials and its joint. Hence In this project effect of hydrogen on AA6061 and its joint with SS304 has been evaluated using different mechanical and microscopic studies.

### **Chapter 3**

## **Research Methodology**

In this research work, AA6061 alloy has been chosen to investigate the effect of hydrogen embrittlement which is a major issue due to exposure in hydrogen because now-a-days it is seen as a future fuel. And due to the hybrid modification of structures, its joint properties with SS304 which is to be obtained through solid state welding technique i.e. Friction Stir Welding also needs to be studied when exposed to hydrogen as a fuel tank, supply pipelines etc.

- For AA6061 alloy: The required samples for experiment were obtained through fabricated sheets of AA6061 followed by shearing operation. Then from it 5mm x 5mm samples and dog bone specimen for tensile test were obtained through wire EDM. The obtained samples were electrochemically charged with hydrogen under different time intervals. Then samples were taken for metallography. After that material characterization was done i.e. microstructure and hardness. Then the samples were taken for mechanical testing i.e. tensile test and hardness, to evaluate the mechanical properties. Further XRD and SEM was also done.
- For AA6061-SS304 weld joint: SS304 plates were welded with AA6061 plates of dimensions 200mm x 50 mm x 3mm. The obtained samples were electrochemically charged with hydrogen under different time intervals. Then from it 5mm x 5mm samples and dog bone specimen for tensile test were obtained through wire

EDM. The obtained samples were then taken for metallography. After that material characterization was done i.e. microstructure and hardness. Then the samples were taken for mechanical testing i.e. tensile test and hardness, to evaluate the mechanical properties.

### **Chapter 4**

## **Research Objectives**

Now-a-days hydrogen is seen as a future fuel in vehicle. So it becomes important to store it safely since it causes hydrogen embrittlement and affects the properties of the material significantly. Studies suggested, techniques to evaluate the mechanical properties with respect to fusion state welding but fewer evaluation of the properties with respect to solid state welding technique in AA6061-SS304 was done. The objective is to analyse the mechanical properties of AA6061 alloy under different time intervals when charged with hydrogen. Along with it, the mechanical properties of AA6061-SS304 weld joint, when electrochemically charged with hydrogen was also studied which was obtained through Friction Stir Welding which is one of the solid state welding technique.

### **Chapter 5**

## **Experimental Details**

#### **5.1 Material Preparation**

- For AA6061 Alloy: The specimens of dimensions 5mm x 5mm was cut by wire EDM from the respective plate of 50mm x 50mm which was obtained from its respective sheet by shearing machine. The obtained specimen was Samples for tensile test were also obtained form that 50mm x 50mm plate.
- For AA6061-SS304 Weld Joint: 50mm x 50mm plates of AA6061 and SS304 were joined by Friction Stir Welding Process. Then from it, samples were cut for tensile test and 5mm x 5mm for research purpose.

#### **5.2 Microstructure and Characterizations**

The obtained specimens of 5mm x 5mm were mounted with the help of hot mounting machine. Then the obtained samples were thoroughly polished by polishing machine with AMRI papers of grades 400, 600, 800, 1000, 1200, 1500 and 2000. After that that diamond polishing was performed by applying diamond paste and lubricating agent was applied time-to-time. After obtaining the final specimens respective etchant was applied to obtain the microstructures.

The microstructures were obtained with the help of Optical Microscopeand the respective microstructures are shown in their respective sections.

#### 5.3 Welding

AA6061 and SS304 plates of dimensions 200mm x 50mm of 3mm thickness were welded together by Friction Stir Welding technique with the help of ELLIOT STURDMILL 1200. The figure in their respective section shows the Friction stir welding machine setup and the specimen so obtained after welding.

#### **5.4 Mechanical Properties**

The mechanical properties such as tensile strength was measured with the help of tensile testing machine, the hardness of the sample was obtained by Vickers Hardness testing machine. Futher, SEM and XRD was also carried out.

Mechanical properties such as yield strength (YS), ultimate tensile strength (UTS) and elongation (%) were obtained to study the effects of hydrogen embrittlement on AA6061 and AA6061-SS304 weld joint.

### **Chapter 6**

## **Results and Discussion**

In this chapter, effects of charging hydrogen in AA6061 alloy and weld joint of AA6061-SS304 obtained through Friction Stir Welding has been investigated. The effect of hydrogen embrittlement in the above specimens has been observed by evaluating their mechanical properties.

#### **6.1 Specimen Preparation**

• For AA6061 alloy: The set-up shown below shows the Electrochemical charging Instrument. Hydrogen is induced in the specimens through electrochemical charging process at different time intervals through a DC power supply.(About machine). The electrolyte was composed of 75% methanol, 22.4% DI water and 2.6% H<sub>2</sub>SO<sub>4</sub> (conc.) and then the obtained solution was further poisoned with AS<sub>2</sub>O<sub>3</sub> (0.1gm/l). Here, AS<sub>2</sub>O<sub>3</sub> acts as hydrogen recombination inhibitor. The specimen was used as cathode and the graphite electrode was used as anode. Then the specimens were charged under this atmosphere at 0.05 A and 2.8 V. The specimens so obtained was then used for the further evaluation of the properties.

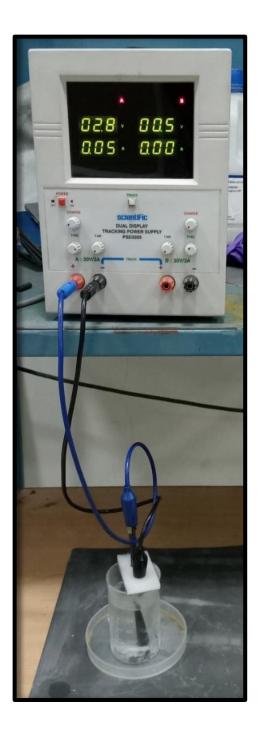


Figure 6.1: Eletrochemical Charging Setup

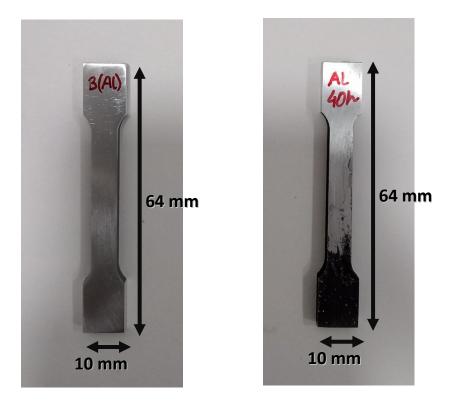


Figure 6.2: Hydrogen charged specimen for tensile test



**Figure 6.3:** Hydrogen charged specimens for Microstructure, Hardness and XRD analysis

• For AA6061-SS304 Weld Joint: AA6061 and SS304 plates of dimensions 200mm x 50mm of 3mm thickness were welded together by Friction Stir Welding technique (ELLIOT STURDMILL 1200). The figure shown below shows the Friction stir welding machine setup and the specimen so obtained after welding.



Figure 6.4: Friction Stir Welding Machine

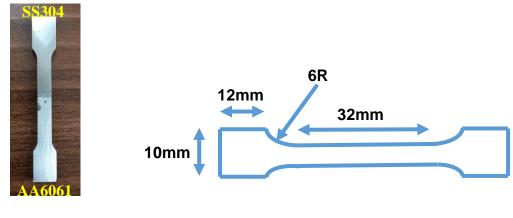


Figure 6.5: Setup showing plates about to be welded



Figure 6.6: Welded plates

Then the required samples were cut form it to evaluate its mechanical properties.

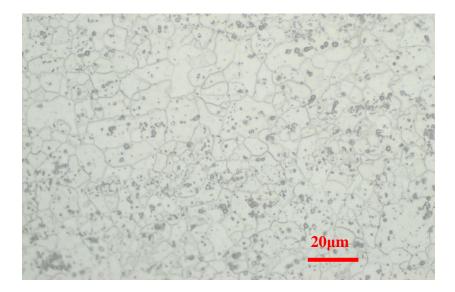


**Figure 6.7:** Tensile test specimen of AA6061-SS304 weld joint

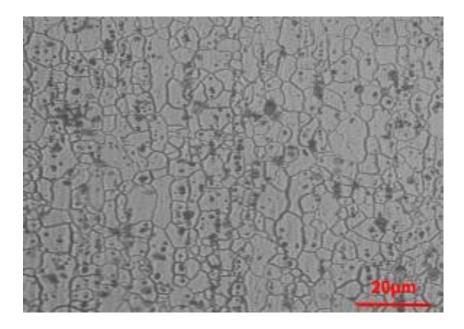
**Figure 6.8:** Dimensions of tensile test specimen of AA6061-SS304 weld joint

## 6.2 Microstructural Analysis

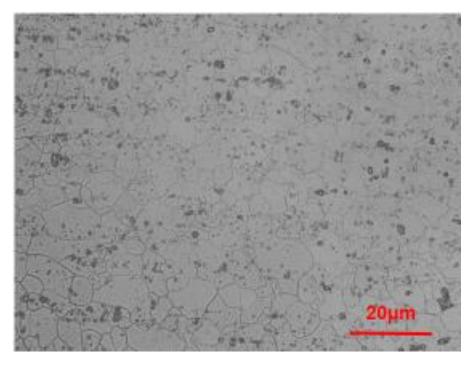
Fig 6.9 below illustrates the microstructure of the base as well as hydrogen charged specimens which was observed under the Optical microscope (ZEISS Axiocam 503 Color).



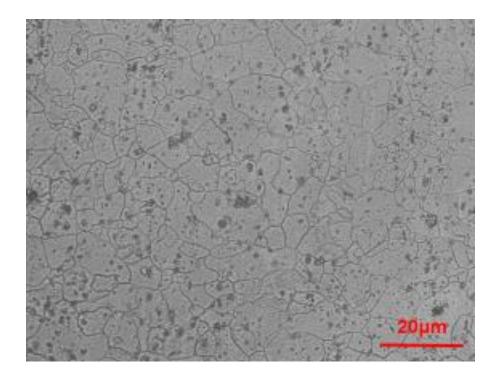
(a)



(b)



(c)



(d)

Figure 6.9: Microstructure of charged specimens at 20x of (a) Base (b) 40h

(c) 80h (d) 140h

### 6.3 Tensile Test

• For AA6061 alloy: Tensile test was conducted on the specimens by tensile testing machine (Manufactures by BISS). The figure below show the dimensions of the tensile test specimen.

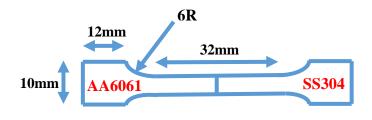


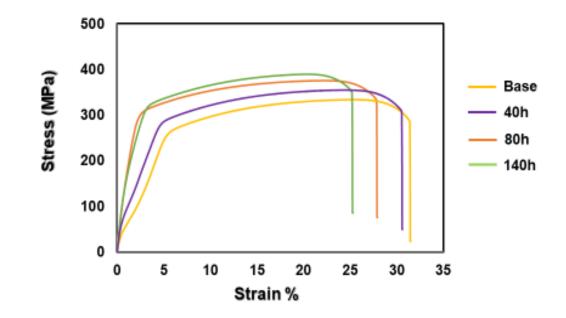
Figure 6.10: Dimensions of the tensile test specimen.

The table 6.1 below shows the variation of the tensile strength of the material under different charging times.

**Table 6.1:** Variation of strength and elongation % with respect to charging time for AA6061 alloys

SAMPLE ID	GAUGE LENGTH (in mm)	WIDTH (w) (in mm)	Thickne ss (t) (in mm)	Ultimat e Tensile Strengt h (σut) (in MPa)	Elongatio n (in %)	Yield Streng th (σyt) (in MPa)
AA6061 (Base)	25	6	3	333.524	20.607	183.30
AA6061 (40h)	25	6	3	354.596	29.476	210.80
AA6061 (80h)	25	6	3	375.305	27.99	170.18
AA6061 (140h)	25	6	3	389.232	25.298	170.70

From the above table it can be observed that as the charging time is increased the strength of the material is also getting increased. Then from the above data a graph was plotted between stress and strain % to observe the variation of the brittleness nature of the specimens. The respective graph is shown below.



**Figure 6.10:** Graph showing the variation of tensile strength and strain % of hydrogen charged specimens for different charging time intervals for AA6061

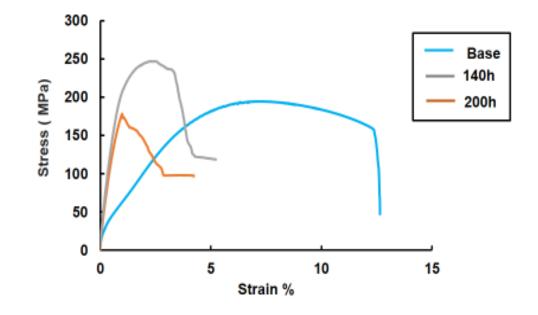
The increase in strength is due to the formation of the intermetallic compound as Aluminium hydride i.e. AlH<sub>3</sub>.

• <u>For AA6061-SS304 Weld Joint:</u> Tensile test was conducted on the specimens by MACHINE NAME. The table 6.2 below shows the variation of the tensile strength of the material under different charging times.

**Table 6.2:** Variation of strength and elongation % with respect to chargingtime for AA6061-SS304 weld joint.

SAMPLE ID	GAUGE LENGTH (in mm)	WIDTH (w) (in mm)	Thickness (t) (in mm)	Ultimate Tensile Strength (σut) (in MPa)	Elongation (in %)	Yield Strength (o <sub>yt</sub> ) (in MPa)
AA6061- SS304 (Base)	25	6	3	194.423	12.6	79.83
AA6061- SS304 (140h)	25	6	3	178.10	4.4	154.352
AA6061- SS304 (200h)	25	6	3	246.767	1	189.433

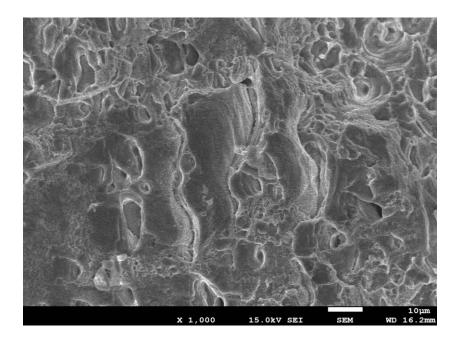
From the above table it can be observed that as the charging time is increased the strength of the material is also getting increased. Then from the above data a graph was plotted between stress and strain % to observe the variation of the brittleness nature of the specimens. The respective graph is shown below.



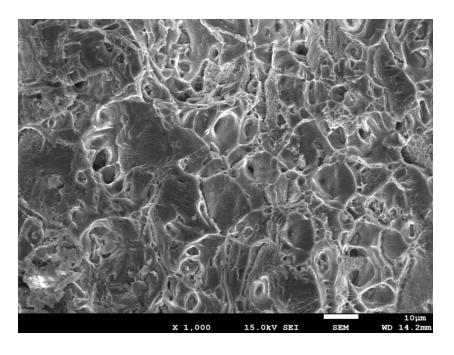
**Figure 6.11:** Stress-strain curve of hydrogen charged specimens for different charging time intervals for AA6061-SS304 weld joint

### 6.4 SEM

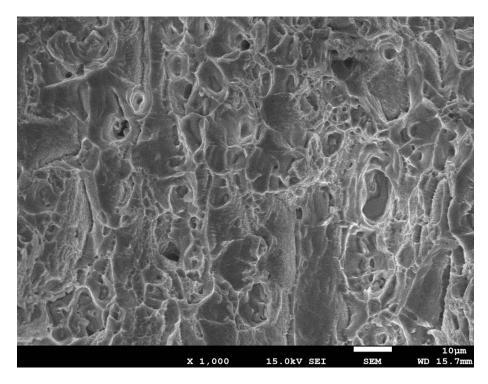
• For AA6061 alloy: The fractured surfaces obtained after tensile test results were observed under Scanning Electron Microscope for different hydrogen charged specimens. The results obtained so are shown below.



(a)



(b)



(c)

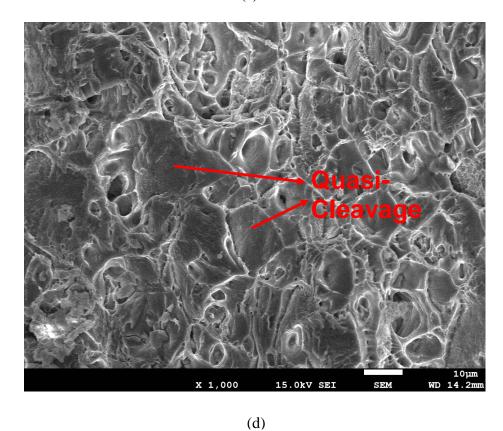


Figure 6.12: Fractured Surfaces observed under SEM of (a) Base

(b) 40h (c) 80h (d) 140h

This quasi-cracked and intergranular fracture formation is attributed to the fact due to the transition of the specimen from the ductile to brittle nature due to hydrogen charging.

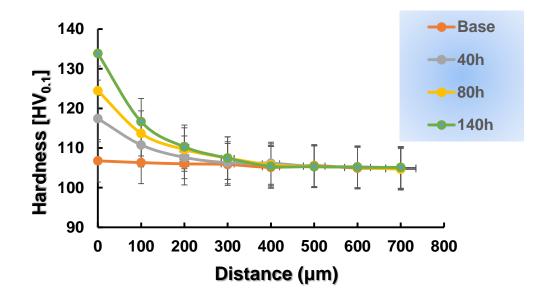
### **6.5 Hardness Test**

• For AA6061 alloy: Hardness test was conducted on the specimens by Vickers hardness tester. The table below shows the variation of hardness with increasing depth for different specimens.

SAMPLE ID	AVERAGE VICKERS HARDNESS NUMBER		
	(HV <sub>0.1</sub> )		
AA6061 (Base)	105.84		
AA6061 (40h)	111.19		
AA6061 (80h)	114.53		
AA6061 (140h)	119.47		

Table 6.3: Variation of Hardness of AA6061 with charging time

Then the graph was plotted between hardness and depth as shown below.



**Figure 6.13:** Vickers Hardness vs Depth curve of hydrogen charged specimens for different charging time intervals for AA6061 alloy

The hardness increases with the increase in charging time and for a constant charging time the hardness decreases with increasing depth till it attains a constant value.

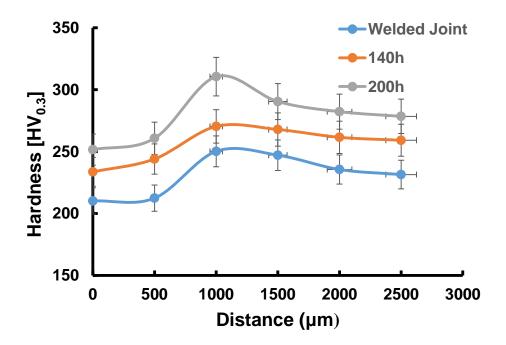
The increase in hardness was attributed to the fact that the solute hydrogen atoms acts as dislocation pinning sites which increased the surface hardness of the Aluminium alloy.

• For AA6061-SS304 weld joint: Hardness test was conducted on the specimens by Vickers hardness tester. The table below shows the variation of hardness with increasing depth for different specimens.

**Table 6.4:** Variation of Hardness of AA6061-SS304 weld jointwithcharging time

SAMPLE ID	AVERAGE VICKERS HARDNESS NUMBER		
	(HV <sub>0.1</sub> )		
AA6061-SS304 (Base)	247.07		
AA6061-SS304 (140h)	267.78		
AA6061-SS304 (200h)	290.33		

Then the graph was plotted between hardness and depth as shown below.



**Figure 6.14:** Vickers Hardness vs Depth curve of welded charged specimens for different charging time intervals.

### 6.6 XRD

• <u>For AA6061 alloy:</u> XRD was performed on the hydrogen charged specimens and the data so obtained is plotted as shown below.

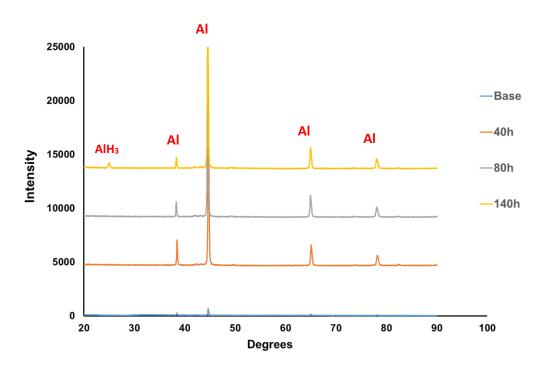


Figure 6.15: XRD plot of AA6061

The presence of Aluminium hydride (AlH<sub>3</sub>) was a result of the increased hydrogen concentration which exceeded the solubility of hydrogen in the Aluminium alloy.

## Chapter 7

# **Conclusions and Scope for Future Work**

### 7.1 Conclusions

The project work has been carried out on AA6061 and AA6061-SS304 weld joint by FSW. The present studies suggests that:

- For AA6061 Alloy: As the charging time is increased,
  - The tensile strength of the material also gets increased.
  - > The hardness of the material also gets increased.
  - The microstructural changes also suggests that for charging it for 140 hours it shows some changes.
  - SEM results shos formation of quasi-cleavage which shows the transition of the material from ductile to brittle.
  - XRD study reveals that for charging time of 140 hours hydrogen is present in it in the form of Aluminium hydride (AlH<sub>3</sub>).
- For AA6061-SS304: As the charging time is increased,
  - The tensile strength of the material also gets increased.
  - > The hardness of the material also gets increased.

### 7.2 Future Scope

- This project gas been carried out by varying one parameter as time so far. But other process parameters can also be varied to study the effect of hydrogen embrittlement.
- Since the components are also subjected to cyclic loading conditions so it also becomes important to study and observe the fatigue properties of the components to ensure the safe working of the components.

### REFERENCES

[1] Schwankl, M., Wedler, J., and Korner, C. (2016). Wrought Al-Cast Al compound casting based on zincate treatment for aluminum wrought alloy inserts. Journal of Materials Processing Technology, 238, 160-168.

[2] Madhav, B. V., Mohiuddin, A. A., and Atifuddin, M. (2016).
 Comparison of mechanical properties of AlSiMg alloy with varied composition. International Journal of Engineering and Technology Research, 4 (4), 12-19

[3] Sinyavskii, V. S., and Kalinin, V. D. (2005). Marine corrosion and protection of aluminum alloys according to their composition and structure. Protection of metals, 41(4), 317-328.

[4] Tan, E., and Ogel, B. (2007). Influence of heat treatment on the mechanical properties of AA6066 alloy. Turkish Journal of Engineering and Environmental Sciences, 31(1), 53-60.

[5] Shin, J., Kim, T., Kim, D., Kim, D., and Kim, K. (2017). Castability and mechanical properties of new 7xxx aluminum alloys for automotive chassis/body applications. Journal of Alloys and Compounds, 698, 577-590.

[6] Joseph, O. O., & Babaremu, K. O. (2019). Agricultural Waste as a Reinforcement Particulate for Aluminum Metal Matrix Composite (AMMCs): A Review. Fibers, 7(4), 1-9.

[7] CAMM Metals Inc. , https://inbound.cammmetals.com/blog/what-are-the-challenges-of-welding-aluminum.

[8] Ambituuni, A.; Amezaga, J.M.; Werner, D. Risk assessment of petroleum product transportation by road: A framework for regulatory improvement. Saf. Sci. **2015**, 79, 324–335.

[9] Fleming, C.H.; Spencer, M.; Thomas, J.; Leveson, N.; Wilkinson, C. Safety assurance in NextGen and complex transportation systems. Saf. Sci. **2013**, 55, 173–187.

[10] Ji, M.; You, X.; Lan, J.; Yang, S. The impact of risk tolerance, risk perception and hazardous attitude on safety operation among airline pilots in China. Saf. Sci. **2011**, 49, 1412–1420.

[11] Greene, D.L.; Ogden, J.M.; Lin, Z. Challenges in the designing, planning and deployment of hydrogen refueling infrastructure for fuel cell electric vehicles. eTransportation **2020**, *6*, 100086.

[12] Michler, T.; Wackermann, K.; Schweizer, F. Review and Assessment of the Effect of Hydrogen Gas Pressure on the Embrittlement of Steels in Gaseous Hydrogen Environment. Metals **2021**, 11, 637.

[13] Kansara, S.; Gupta, S.K.; Sonvane, Y.; Gajjar, P.N. Ultrathin Pd and Pt nanowires for potential applications as hydrogen economy. Mater. Today Commun. **2021**, 26, 101761.

[14] Schaupp, T.; Schroeder, N.; Schroepfer, D.; Kannengiesser, T. Hydrogen-Assisted Cracking in GMAWelding of High-Strength Structural Steel—A New Look into This Issue at Narrow Groove. Metals **2021**, 11, 904. [15] Ajanovic, A.; Haas, R. Prospects and impediments for hydrogen and fuel cell vehicles in the transport sector. Int. J. Hydrogen Energy **2020**, 46, 10049–10058.

[16] Moshtaghi, M.; Safyari, M.; Kuramoto, S.; Hojo, T. Unraveling the effect of dislocations and deformation-induced boundaries on environmental hydrogen embrittlement behavior of a cold-rolled Al–Zn–Mg–Cu alloy. Int. J. Hydrogen Energy **2021**, 46, 8285–8299.

[17] R. J. Gest and A. r. Troiano: Corr., 1974, vol. 30, pp. 274-79

[18] J. Albrecht, B. J. Mctiermann, I. M. Bernstein, and A. W. Thompson: Scripta Metall., 1977, vol. 11, pp. 893-97.

[19] G. M. Scamans: Jour. Mat. Sci., 1978, vol. 13, pp. 27-36.

[20] J. Albrechet, A. W. Thompson, and I. M. Bernstein: Metall. Trans. A, 1979, vol. 10A, pp. 1979-66.

[21] L. Christodoulou and H. M. Flower: Acta Metall., 1980, vol. 28, pp. 481-87.

[22] D. A. Hardwick, M. Taheri, A. W. Thompson and I. M. Berstein: Metall. Trans. A, 1982, vol. 13A, pp 235-39.

[23] C. Dharmendra, K. P. Rao, J. Wilden, S. Reich, Study on laser welding-brazing of zinc coated steel to aluminum alloy with a zinc based filler, Mater. Sci. Eng. A 528 (2011) 1497-1503.

[24] R. Qiu, C. Iwamoto, S. Satonaka, The influence of reaction layer on the strength of aluminum/steel joint welded by resistance spot welding, Mater. Charact. 60 (2009) 156-159.

[25] L. H. Shah, A. S. M. Yusof, M. Ishak, Investigation of heat treatment on weld joint quality of stainless steel and aluminium dissimilar welding, Proceedings of the 16th International Conference on Advances in Materials & Processing Technologies (AMPT'14) (2013).

[26] M. N. A. Razak, Investigation of heat treatment (preheat) of steel on aluminium-steel welding, Degree thesis, Universiti Malaysia Pahang (2013).

[27] M. Kreimeyer, F. Vollertsen, Gap tolerant joining of aluminium with steel sheets using the hybrid technique, Proceedings of International Congress on Applications of Lasers and Electro-Optics 2006 (ICALEO'06) (2006) 947–952.

[28] H. Bang, H. Bang, G. Jeon, I. Oh, C. Ro, Gas tungsten arc welding assisted hybrid friction stir welding of dissimilar materials Al6061-T6 aluminum alloy and STS304 stainless steel, Mater. Des. 37 (2012) 48–55.