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# ABSTRACT

Nowadays, most of the automotive companies have been searching for new composite materials in order to use on the vehicle body white panes, side impact bars, airbag deployment doors instead of using light steels, also, other sectors such as aerospace, building constructions, transportation.

They have been trying to find new composites for having weight reduction and better shock absorption in case of crush. Aluminium is a good material in terms of having less weight compare to steels, cheaper than other composites materials such as fibreglass and exotic plastics and it has got good corrosion resistance.

For automotive companies, reduction on the weight points that having less fuel consumption which means less CO<sub>2</sub> emission, increase of performance and increase of shock absorption in case of accident. Considering all the good benefits of Aluminium, It is decided to produce light metallic sandwich composite AFS (Aluminium Foam Sandwich) made by Aluminium and Aluminium foam.

The materials (2 or more) inside the sandwich composite give special properties for the final composite. In case of investigation of these final properties, it is necessary to produce the sandwich composite and to do correct tests for analyzing in consideration of each material inside the composite. Aluminium-7046 and Aluminium-foam were selected to use for producing this composite while using pure Zinc and Zinc 2%Aluminium as brazing material in order to join them together. Appropriate brazing conditions were obtained after some experiments and decided that the optimal one is 500 °C and 5 minutes dwell time for Al-7046 with brazing material pure Zn. And the optimal is 520 °C for the one with brazing material Zn2%Al, both of them were produced in argon atmosphere.

6 composite sandwich materials were produced for each 2 groups with pure Zn and Zn2%Al. As following, solution heat treatment was implemented on the parameters of 370°C and 3 hours dwell time, water quench for 3 composite sandwich materials from each 2 groups in order to investigate the benefits of solution heat treatment as following to the brazing heat treatment.

As final step; 3 point bending test, SEM (Scanning Electron Microscope), OM (Optical Microscope), micro-hardness and EDS (Energy Dispersive Spectroscopy) were performed in order to analyze the joints in the microstructure and mechanical properties.

Also, all these tests on above were implemented to the Al-7046 as received plates, Al-7046 plates after brazing heat treatment and Al-7046 plates after solution heat treatment for analyzing the effects of the heat treatments in terms of mechanical properties and microstructures.

The focus of this experimental project is to find best optimal joining conditions and optimal mechanical properties of AFS products.

# CHAPTER 1.BACKGROUND

## 1.1 Aluminium

The particular composition of aluminium properties and its alloys provide aluminium one of the most all-purpose, economical, and interesting metallic materials for a huge range of soft and ductile engineering applications. After the use of steels, aluminium alloys takes the second place as structural metals [1].

The quantity of the Aluminium in the earth is plentiful. It is always seen in a compound structure both with some of its minerals being cryolite, turquoise, corundum, alunite, bauxite, and such silicates as kaolin and mica. The metal aluminium is produced from the raw material of Bauxite a ferruginous aluminium hydroxide. Chemical processes are applied to bauxite in order to eliminate impurities for obtaining alumina. Alumina or aluminium oxide has got looking like refined sugar with hard crystalline body. Electrolyzing solution of alumina in molten fluorides helps us to obtain aluminium [2].

Aluminium has good strength, high thermal and electrical conductivity, good reflectivity, high corrosion resistance, good workability, and interesting appearance also is light weight material. At the same time all kind of finish can be obtained with its non-magnetic, non-toxic, and non- sparking features. In Table 1 aluminium properties and its alloys are shown [3].

Table 1: Aluminium properties [3]

Tensile Strength, ksi (MPa)	10-72 (70-505)
Yield Strength, ksi (MPa)	3-65 (20-455)
Elongation %	<1-30
Hardness, HB	30-150
Electrical Conductivity, %IACS	18-60
Thermal Conductivity, Btu.in./h.ft <sup>2</sup> .°F at 77 °F (W/m.K at 25 °C)	660-1155 (85-175)
Fatigue limit, ksi (Mpa)	8-21 (55-145)
Coefficient of Linear Thermal Expansion at 68-212°F (20-100°C)	9.8-13.7 x 10 <sup>6</sup> / °F
Shear Strength, ksi (Mpa)	6-46 (42-325)
Modulus of elasticity, 10 <sup>6</sup> psi (Gpa)	9.5-11.2 (65-80)
Specific Gravity	2.57-2.95

### **1.1.1 Applications of Aluminium**

In the beginning, aluminium applications were narrow with house numbers, hand mirrors, combs, brushes, tie clasps, cuff links, hat pins, and decorative lamp housings that reason of having light weight, silvery finish, and novelty of the new metal. Cast aluminium cookware was alternative usage to cast iron and brass pots, pans, and kettles. The price of aluminium continuously decreased and important engineering applications became economically feasible [3].

Three rising sectors came across with the existence of aluminium; for electrification propose aluminium has got low-density, good corrosion resistant, high-conductivity wire and cable for which aluminium was well-suited, on the other hand, also for transmission towers and cast installation hardware. Automotive pioneers needed to innovative materials and product forms to differentiate the performance and appearance of their products. First time, Wright Brothers succeeded in powered flight, engine and other parts in cast aluminium showed the starting of researching and applying aluminium in the aerospace industry [3].

Cast aluminium has been applied perfectly in power-train applications such as engine blocks, cylinder heads, pistons, transmission cases, and oil pans for the reason of having light weight in order to get better on fuel efficiency, that improvement in the mission of cast aluminium obliged replacement in more important structural parts needing the standards of new component designs, materials, and production techniques. Aluminium castings will take a important place in the future when unavoidable electric, hybrid technologies are improved to join components, design and construction techniques for maximum efficiency [3].

Other general applications are as following; architectural panels and spandrels, outdoor lamp housings, lawn mower decks, marine components, cooking utensils, parts used in food, dairy, beverage processing, medical and dental equipment, electronic cabinet frames and components, tire moulds, escalator and moving sidewalk tread plates parts, highway railing posts, machinery, transmission cases, gas meters and regulators, gear blocks, gear cases, fuel pumps, impellers, instrument cases, lawnmower decks, intake manifolds, cylinder heads, clutch housings, outboard motor propellers, automotive space frames, automotive wheels, truck wheels, axle and differential housings, meter bodies, compressor bodies, dies for plastic injection moulding, suspension saddles, aircraft pylons, canopies, flaps, speed-brakes hatch covers, wave guides, electronic cases, missile bodies, fins, industrial beam heads, brake cylinders, engine cooling fans, clutch housings, crankcases, structural aerospace components, air compressor pistons, fuel pumps, compressor cases, rocker arms, timing gears, cylinder heads, gear housings, cylinder liners, marine engines, tooling plate, explosion-proof enclosures, electrical fittings, brazed parts, pistol frames, decorative parts, reflectors, optical systems, brake valves, hydraulic pumps, steering column parts, airbag containment housings, power brake proportioning valves, bicycle frames, scraper blades and shoes, sporting equipment, golf club heads, aerospace optical systems, electronic/avionic thermal management components, pressure vessels, brackets [3].

## 1.2 Aluminium Foam

Metal foams are a new type of materials with low densities and new physical, mechanical, thermal, electrical and acoustic properties. They provide big potential for lightweight structures both for energy absorption and thermal management; and some of them, at least, are cheap. They are also both recyclable and nontoxic [4].

Applications of Aluminium foams have huge importance in the automotive industry, which has been extremely interested in metal foams since they were first developed. Potential applications also exist in shipbuilding, the aerospace industry, and civil engineering [5].

Table 2: Typical properties of three families of Aluminium foams [5]

	Gas-Injection Foams	Blowing-Agent Foams	Powder-Compaction Foams
<b>Typical End Products</b>	Panels: $\leq 16\text{m} \times 1\text{m} \times 0.2\text{m}$ Complex-shaped parts	Blocks: $\leq 2\text{m} \times 0.6\text{m} \times 0.5\text{m}$ Slices: 10 mm thick	Blocks: $\leq 1\text{m} \times 0.5\text{m} \times 0.2\text{m}$ Complex-shaped parts Sandwich panels: $\leq 2\text{m} \times 1\text{m} \times 0.03\text{m}$
<b>Density Range (g/cm<sup>3</sup>)</b>	0.069-0.54	0.18-0.24	0.3-0.7
<b>Pore Diameter (mm)</b>	3-25	2-10	2-10
<b>Cell Wall Thickness (<math>\mu\text{m}</math>)</b>	50-85	...	50-100
<b>Alloy Range Available</b>	Al alloys	Al, AlZnMg	Al-, Zn-, Pb-, Sn-, Au- alloys

The properties of metal foam and other cellular metal structures depend upon the properties of the metal, the relative density and cell topology (e.g. open or closed cell, cell size, etc.) [4].

Main production methods of Aluminium foams [4];

1. Bubbling gas through molten Al–SiC or Al–Al<sub>2</sub>O<sub>3</sub> alloys.
2. By stirring a foaming agent (typically TiH<sub>2</sub>) into a molten aluminium alloy and controlling the pressure while cooling.
3. Consolidation of a aluminium powder with a particulate foaming agent (TiH<sub>2</sub> again) followed by heating into the mushy state when the foaming agent releases hydrogen, expanding the material.

As explained before, cell topology is divided mainly into two groups, closed cell and open cell. The pores of closed cell aluminium foams are closed with cell membranes, whereas there are no cell membranes along the surface for open cells, thus voids are interconnected.

### 1.3 Aluminium Foam Sandwich (AFS)

Aluminium Foam Sandwich (AFS) is a product comprising a highly porous aluminium alloy foam core and two aluminium alloy face sheets. The layers are firmly attached to each other by metallic bonding. Use of such sandwich panels has been proposed for many industrial sectors including automotive, ship building, railway and aircraft industry. Sandwich panels compared to dense material or bare foam have various advantages. They are stiffer than dense sheets of equal mass. Compared to bare foam without face sheets the main advantage is that the outer skin allows the sandwich to bear tensile loads that occur, e.g. when the panel is bent [6].

Two car components have a major impact on fuel consumption: on the one hand, the drivetrain, and the car body, with the parameters aerodynamics and weight. Whereas aerodynamics was the main topic in last year's body development, lightweight body design is now the target of many research programs. Increasing demands in car safety and vehicle dynamics have constantly been pushing up the car mass. Especially measures to improve torsion stiffness contribute to this trend. This is even more significant, as the body structure is weakened due to concept features with respect to convertibles. To find the solution to the target conflict of high body stiffness at low weight, led to use of the sandwich panel principal. The first effort to implement sandwich structures was stopped by the lack of economical ways to manufacture these mostly three-dimensional shaped parts. In a second step investigations in metal foams took place, especially in aluminium foams. Because of its manufacturing process aluminium foam is well suitable for complex shaped parts and energy absorbing components [7].



Figure 1: Aluminium foam sandwich (AFS)

As shown on the figure 1, AFS composite which I produced and will be discussed later, there are two layers of Aluminium 7046 plates on the surfaces and in between highly porous Aluminium Foam.

### **1.3.1 Applications of Aluminium Foam Sandwich (AFS)**

#### **Electromagnetic Shielding**

AFS material has a high stiffness-to-mass ratio. Therefore it is well suited for building light-weight structures that can bear loads while their deformation remains small. AFS has the additional useful functionality that it shields electromagnetic waves very well. Electromagnetic damping values as good as  $-118$  dB were measured in the frequency range from 100 to 500 MHz for a box made of 15 mm AFS with a core density of  $0.48 \text{ g cm}^{-3}$ . A box made of aluminium sheet of the same weight exhibited a damping value of  $-57$  dB [26]. Therefore, AFS is a good candidate for electromagnetic damping, mechanical rigidity and electrical conductivity at the same time [6].

#### **The Rear Close-Out-Panel**

Between trunk and rear seats has been chosen as an example part for developing the production technology. The AFS component uses the same part layout as the steel panel; the package space requirements have been increased by only 3 mm. The weight has been reduced by 25% whilst the stiffness reached is seven times that of the steel stamped original [7].

#### **Aluminium Foam Car Body Structures**

Aluminium foams offer cost-effective performance as structural automotive parts that are up to ten times stiffer and 50% lighter than equivalent parts made of steel. Such lightweight, stiff foam sandwich panels simplify body structure systems. The three-dimensional aluminium foam system consists of outer skins roll-bonded to a central layer containing a dispersion of titanium hydride. The central section is expanded by heat treatment after the panel has been pressed to shape. Sections of the body shell considered well suited for aluminium foam sandwich panels include firewalls, roof panels and luggage compartment walls. As much as 20% of the auto structure could be made from three dimensional aluminium foam panels [4].

#### **Bicycle Crank Arm Prototype**

AFS forging has found a first prototypical application with a crank arm for racing bicycles. The lightest parts on the market weigh slightly more than 300 g. The forged AFS replacement of the crank arm, weighs 222 g, i.e. 30% less. This is a big achievement since the lightest products on the market currently differ by some tens of grams only [8].

#### **Cooking**

AFS are useful materials for cooking equipment due to the characteristics of thermal conduction. A point-like heat source directed onto one side of the AFS, will first heat up the face sheet. Since the heat conductivity of the dense face sheet is 30 times higher than that of the foam, heat will be first conducted across the sheet. Diffusion of heat through the foam core will take place at a lower but still high enough rate. After arriving at the opposite sheet, heat will be dissipated in the plane of the face sheet plane again. AFS are therefore efficient heat diffusers [6].

## **Optical Systems for Space Applications**

Mirrors of all sorts play a key role in space technology. Whether the frequencies are optical, infrared or microwave, precision, low mass and freedom from long-term distortion are primary design constraints. The mirror face is stiffened by bonding it to a foam layer backed by a stiff backing plate. The structure is light and very stiff; the all-metal construction gives freedom from long-term distortion [4].

## **Solar Thermal Energy Generation**

It is a technology for harnessing solar energy for heat production and eventually generation of electricity. One possible concept for a parabolic mirror involves AFS that can be bent to the large radii required (about 20 m) with low tolerances. A thin reflecting foil on top of the AFS trough would provide the high reflectivity needed. AFS is a suitable material since it is polymer-free. Unlike polymers, large amplitudes in temperature from + 60°C to 0° from day to night time would not affect AFS and a long lifetime is ensured. Thermal warping is small. The high stiffness-to-mass ratio is favourable to lower deformations caused by the wind and to ease moving the mirror [6].

## **Protection against Blasts, Bullets, Other Hazards**

Combination of AFS with a stone plate has proved to be efficient to stop bullets. The hard stone front plate fragments the bullet and the following aluminium sheets, foam on the back stop the fragments and dissipate energy. The AFS sheet is a convenient light carrier material and allows for fixing the structure. The stone is the material that can be displayed. The anticipated use is on or in buildings such as banks or in public building under threat. As there is little ageing and corrosion in AFS structures, outdoor use is possible without any problems [6].

## **Light Weight Conformal Pressure Tanks**

Fluids in pressure tanks, ranging from aircraft fuel to liquid nitrogen cryogen for airborne infrared telescopes, must be maintained at constant and uniform temperature, uniform pressure, and in moving systems must be restrained from 'sloshing' because this generates pressure gradients. The foam has a low density, occupying only 10% of the volume of the tank, but it limits fluid motion. The high thermal conductivity of the cell edges maintains the fluid at a constant temperature, important for jet fuel [4].

# CHAPTER 2. LITERATURE SURVEY

## 2.1 Wrought Aluminium Alloys-Aluminium 7046

It is convenient to divide aluminium alloys into two major categories: wrought compositions and cast compositions. A further differentiation for each category is based on the primary mechanism of property development. Many alloys respond to thermal treatment based on phase solubility. These treatments include solution heat treatment, quenching, and precipitation, or age, hardening. For either casting or wrought alloys, such alloys are described as heat treatable. A large number of other wrought compositions rely instead on work hardening through mechanical reduction, usually in combination with various annealing procedures for property development. These alloys are referred to as work hardening [1].

For wrought alloys a four-digit system is used to produce a list of wrought composition families as follows [1]:

- 1xxx: Controlled unalloyed (pure) composition, used primarily in the electrical and chemical industries
- 2xxx: Alloys in which copper is the principal alloying element although other elements notably magnesium, may be specified. 2xxx series alloys are widely used in aircraft where their high strength is valued
- 3xxx: Alloys in which manganese is the principal alloying element, used as general-purpose alloys for architectural applications and various products
- 4xxx: Alloys in which silicon is the principal alloying element, used in welding rods and brazing sheet
- 5xxx: Alloys in which magnesium is the principal alloying element, used in boat hulls, gangplanks, and other products exposed to marine environments
- 6xxx: Alloys in which magnesium and silicon are the principal alloying elements, commonly used for architectural extrusions and automotive components
- 7xxx: Alloys in which zinc is the principal alloying element, used in aircraft structural components and other high-strength applications. The 7xxx series are the strongest aluminium alloys, with yield strengths  $\geq 500$  MPa ( $\geq 73$  ksi) possible
- 8xxx: Alloys characterizing miscellaneous compositions the 8xxx series alloys may contain appreciable amounts of tin, lithium, and/or iron

Wrought alloys that constitute heat-treatable (precipitation-hardenable) aluminium alloys include the 2xxx, 6xxx, 7xxx, and some of the 8xxx alloys. The various combinations of alloying additions and strengthening mechanisms used for wrought aluminium alloys are reported in Table 3. The strength ranges achievable with various classes of wrought alloys are given in Tables 4 [1].

Table 3: Classification of wrought Al-Alloys according to their strengthening mechanism [1]

Alloy System	Aluminium Series
<b>Work-hardenable alloys</b>	
Pure Al	1xxx
Al-Mn	3xxx
Al-Si	4xxx
Al-Mg	5xxx
Al-Fe	8xxx
Al-Fe-Ni	8xxx
<b>Precipitation-hardenable alloys</b>	
Al-Cu	2xxx
Al-Cu-Mg	2xxx
Al-Cu-Li	2xxx
Al-Mg-Si	6xxx
Al-Zn	7xxx
Al-Zn-Mg	7xxx
Al-Zn-Mg-Cu	7xxx
Al-Li-Cu-Mg	8xxx

As we can see from the table 3, Al-7046 wrought alloy is in the group of precipitation hardenable alloys category. Thus, we can use some heat treatments such as brazing heat treatment, solution heat treatment and quenching in order to change and improve its mechanical properties. Also, we can see briefly the composition of the 7xxx alloys that it consists of mainly zinc then some small percentages of magnesium and or copper.

The selection reason of Al-7046 for my experimental work is they are the strongest Al alloys in terms of yield strengths and so on. For the applications of Al-7046 in automotive, aerospace and other sectors, high strengths takes a major part in the applications respecting the weight reduction comparing to the steal or iron based materials.

Another important point is also production of the AFS composite with Al-7046 would give good yield and tensile strengths at the same time using Al-foam material between these 2 layers of Al-7046 would give really good features of energy and shock absorbing in case of forces which applies on the surface of the Al-7046 sheets. Usage of the Al-foam also makes our AFS composite really light in terms of comparing the other materials such as steal in order to get the same mechanical properties.

Table 4: Strength ranges of various wrought aluminium alloys [1]

Aluminium Association Series	Type of alloy composition	Strengthening method	Tensile strength range	
			Mpa	ksi
1xxx	Al	Cold work	70-175	10-25
2xxx	Al-Cu-Mg (1-2.5% Cu)	Heat treat	170-310	25-45
2xxx	Al-Cu-Mg-Si (3-6% Cu)	Heat treat	380-520	55-75
3xxx	Al-Mn-Mg	Cold work	140-280	20-40
4xxx	Al-Si	Cold work (some heat treat)	105-350	15-50
5xxx	Al-Mg (1-2.5% Mg)	Cold work	140-280	20-40
5xxx	Al-Mg-Mn (3-6% Mg)	Cold work	280-380	40-55
6xxx	Al-Mg-Si	Heat treat	150-380	22-55
7xxx	Al-Zn-Mg	Heat treat	380-520	55-75
7xxx	Al-Zn-Mg-Cu	Heat treat	520-620	75-90
8xxx	Al-Li-Cu-Mg	Heat treat	280-560	40-80

As reported on the table 4, 7xxx serial of wrought aluminium alloys have got the highest tensile strength range which makes it useful for weight reduction applications. Another good point which that alloy has got is availability of the heat treatments in order to increase their strength, elongation or elastic modulus. We can apply different kinds of heat treatments depending on our requirements and desire mechanics. For my thesis work, I used two different heat treatment such as brazing heat treatment and solution heat treatment. Briefly saying, the point of using brazing heat treatment was to have good joint between Al-7046 and Al-foam in order to produce AFS composite. As we know in theory that having slow rate of cooling in the heat treatment would decrease the strength of the materials. Thus, after performing brazing heat treatment, another heat treatment was must to do for gaining the strength which we lost. For that case, we need short cooling time which I performed water quench and long dwell time comparing the brazing heat treatment. This new, gaining strength heat treatment is so called solution heat treatment.

7xxx Series, zinc, in amounts of 1 to 8%, is the major alloying element in 7xxx series alloys, and when coupled with a smaller percentage of magnesium results in heat-treatable alloys of moderate to very high strength. Usually other elements, such as copper and chromium, are added in small quantities. Higher strength 7xxx alloys exhibit reduced resistance to stress corrosion cracking and are often utilized in a slightly over-aged temper to provide better combinations of strength, corrosion resistance, and fracture toughness [1].

### 2.1.1 Fabrication of Aluminium and Wrought Aluminium Alloys

To produce pure aluminium, alumina ( $\text{Al}_2\text{O}_3$ ) is first extracted from the mineral bauxite, which contains approximately 50%  $\text{Al}_2\text{O}_3$ . In the Bayer process, a sodium hydroxide solution is used to precipitate aluminium hydroxide, which is then calcined to form alumina. Alumina is then converted to pure aluminium by electrolysis, using the Hall-He'roult process illustrated in Figure 2 [9].

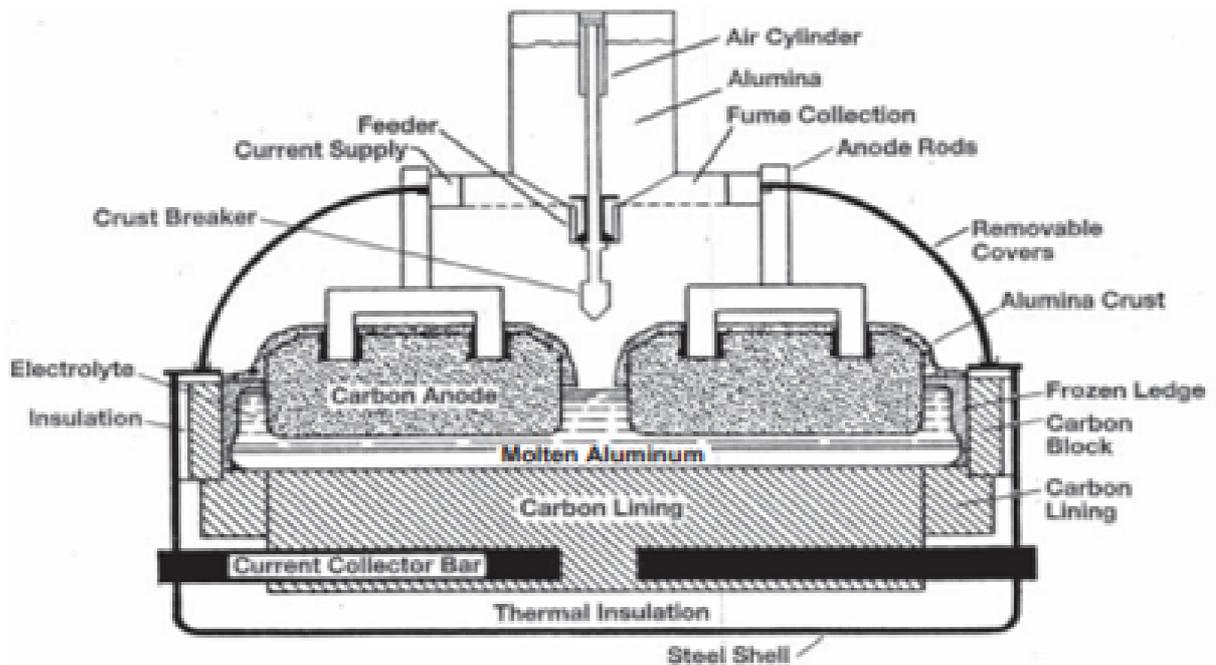


Figure 2: Electrolytic cell used to produce aluminium [9]

The cell is lined with carbon cathodes, and consumable electrodes are gradually fed into the top of the cell. The electrolyte is cryolite ( $\text{Na}_3\text{AlF}_6$ ) with 8 to 10 wt%  $\text{Al}_2\text{O}_3$  dissolved in it. The cell operates at temperatures in the range of 955 to 1010 °C (1750 to 1850 F), with a power rating of 10 to 12 kW.h/kg aluminium. Pure aluminium (99 wt%) is reduced at the cathode and forms a molten pool in the bottom of the cell, which is drained from the bottom and cast into aluminium ingots. Since impurities such as iron and silicon are reduced along with the aluminium, the raw materials used in the Bayer process are carefully controlled to minimize these metal oxides. Since the production of aluminium takes a lot of electrical energy, and recycling aluminium takes much less energy, a large portion of general purpose aluminium currently is made from recycled material [9].

During casting of aluminium alloy ingots, oxides and gases must be controlled. Aluminium oxidizes rapidly in the liquid state and reverts back to alumina. Once the oxide becomes entrapped in the liquid metal, it is difficult to remove and remains dispersed in the liquid metal. The main source of oxygen is moisture from the furnace charge. Oxides are removed by fluxing with gases, solids, or molten salts. Various filtration methods are also used to remove oxides during pouring [9].

Hydrogen is the only gas with appreciable solubility in molten aluminium and can cause blisters in heat treated parts and porosity in aluminium castings. Again, moisture from the furnace charge is the main source of hydrogen. Hydrogen can also be introduced from moisture in the air and products of combustion. Hydrogen is removed by bubbling chlorine, nitrogen, or argon gas through the melt. The semi-continuous direct-chill process is the mostly widely used process for casting commercial ingots that will receive further processing, such as rolling, extrusion, or forging. It produces fine-grained ingots at high production rates. As shown in Figure 3, the molten aluminium is poured into a shallow, water-cooled mould of the required shape [9].

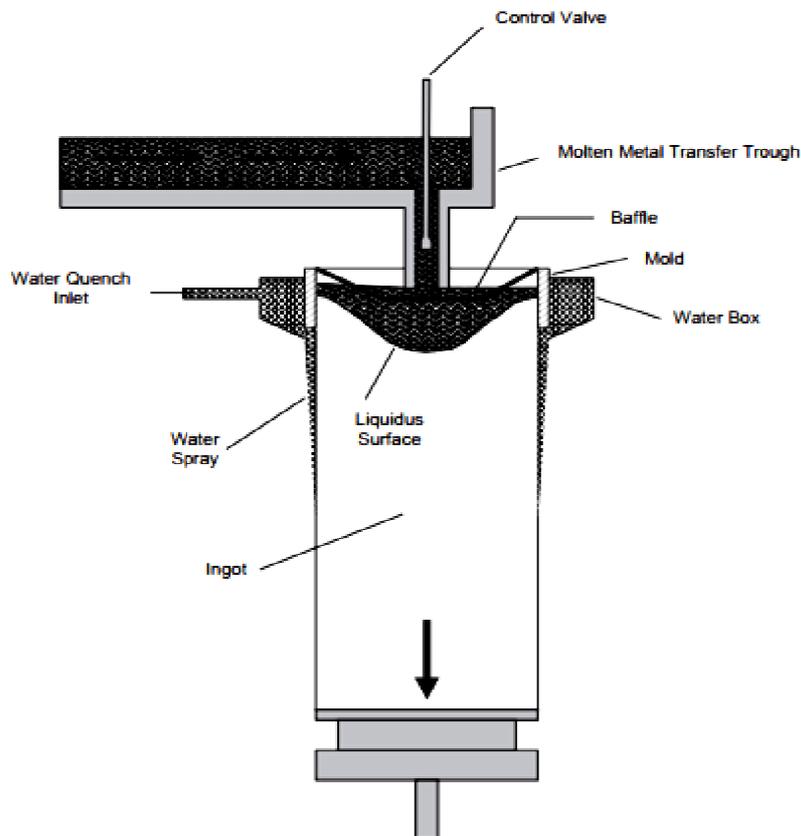


Figure 3: Semi-continuous direct-chill casting [9]

When the metal begins to freeze, the false bottom in the mould is lowered at a controlled rate, and water is sprayed on the freshly solidified metal as it exits the mould. A water box or spray-rings are placed around the ingot to rapidly cool the ingot. Metals with low melting points, such as magnesium, copper, and zinc, are added directly to the molten charge, while high-melting-point elements (e.g., titanium, chromium, zirconium, and manganese) are added as master alloys. Inoculants, such as titanium and titanium-boron, are added to reduce hot cracking and refine grain size. One of the main advantages of direct-chill continuous casting is that it helps to eliminate the segregation that occurs in high-strength alloys produced by the older tilt-casting procedures. These alloys, when produced by tilt casting, are highly segregated because of the broad solidification temperature ranges and the shape of the freezing front. Direct-chill casting eliminates most of this type of segregation because the

liquid metal freezing front is almost horizontal, and the liquid metal freezes from the bottom to the top of the ingot. During direct chill casting, the pouring temperature is maintained at only approximately 28 °C (50 F) above the liquidus temperature; this helps reduce oxide formation and hydrogen pickup and produces a fine-grained structure. It can also produce fairly large ingots at slow speeds, a necessary requirement for the high-strength alloys to prevent cracking. Typical casting speeds are in the range of 2.5 to 13 cm/min (1 to 5 in./min) [9].

### **2.1.1.1 Rolling Plate and Sheet**

Rolled aluminium is the most common of the wrought aluminium product forms. Sheet is defined as rolled aluminium in the range of 0.15 to 6.3 mm (0.006 to 0.250 in.) thick. If the thickness is greater than 6.3 mm (0.250 in.), then it is called plate. Foil refers to aluminium product that is less than 0.15 mm (0.006 in.) thick. Aluminium foil, sheet, and plate are produced from aluminium ingots using the following steps [9]:

1. Scalping of the ingot
2. Preheating and homogenizing the ingot
3. Reheating the ingot, if required, to the hot rolling temperature
4. Hot rolling to form a slab
5. Intermediate annealing
6. Cold rolling along with intermediate anneals to form foil and sheet product forms

Scalping of Ingots; to prevent surface defects on the cast ingot from being rolled into the surface, approximately 6.3 to 9.5 mm (0.250 to 0.375 in.) are removed from the surfaces to be rolled. Some alloys, such as 1100, 3003, and 3015, have fairly smooth as-cast surfaces and do not require scalping. Other, more highly alloyed ingots, such as magnesium-containing alloys and high-strength aircraft alloys, are always scalped [9].

Preheating or homogenizing; puts into solid solution all the constituents that are soluble and reduces the coring that occurs during the casting process. It relieves stresses in the ingot and makes the cast structure more uniform and more readily hot worked. Soaking temperatures and times depend on the specific alloy. Ingots that are going to be clad during hot rolling are scalped after preheating to avoid the heavy oxidation that occurs during the long preheating cycle. This allows the cladding to form a better bond during hot rolling [9].

Hot rolling; conducted above the recrystallization temperature produces a fine grain structure and a minimum of grain directionality. The upper limit is set by the lowest-melting-point eutectic present in the alloy, while the lower temperature is the temperature at which the metal is hot enough to be sufficiently reduced with each pass through the mill without cracking. The ingot is removed from the soaking pit and initially put through a series of four-high rolling mills to break down the ingot structure. Breakdown temperatures are in the range of 400 to

540 °C (750 to 1000 °F), with continuous rolling temperatures of approximately 290 to 455 °C (550 to 850 °F). Since the work rapidly lengthens in the direction of rolling, it is necessary to remove the slab from the mill, turn it around, and then cross roll it to produce wide sheet or plate. The grain structure becomes elongated in the rolling direction, as shown in Figure 4 [9].

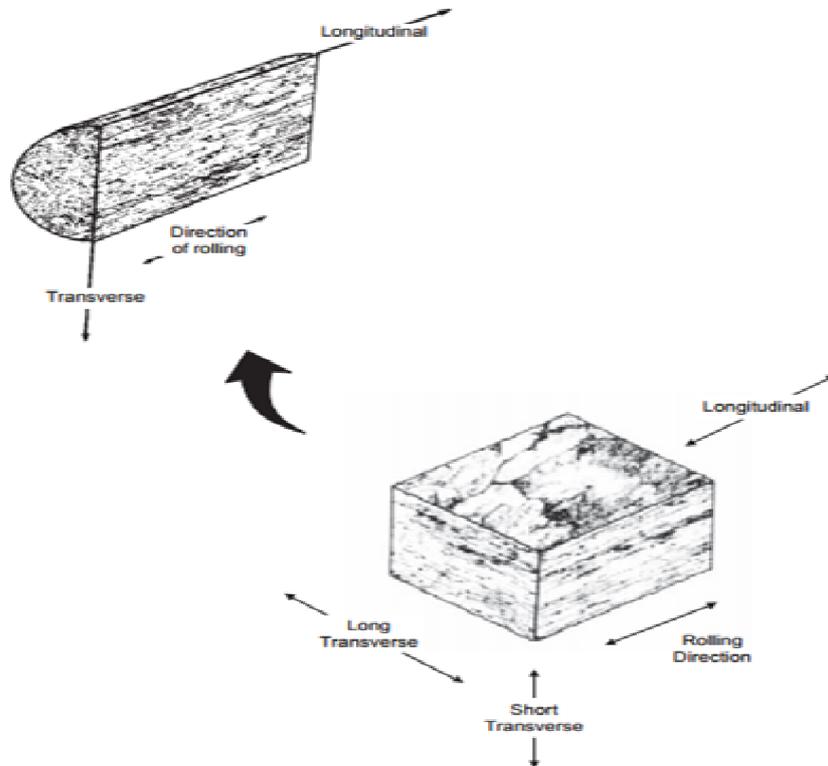


Figure 4: Grain directionality due to rolling. Original magnification at 40x [9]

This results in anisotropic mechanical properties in which the properties are lowest in the through-the thickness (short-transverse) direction. Rolling into thinner plate and sheet is then conducted on five-stand four-high mills, with successive reductions at each mill. Aluminium sheet stock, which can exit the last mill at speeds approaching 485 km/h (300 mph), is coiled into large coils prior to cold rolling [9].

Intermediate Annealing; since hot rolling produces some cold work, coiled aluminium sheet stock is given an intermediate anneal prior to cold rolling. Since the amount of cold work introduced during hot rolling is sufficient to cause recrystallization, annealing not only lowers the strength and it increases ductility, it also imparts a fine grain structure [9].

Cold Rolling; sheet and foil are cold rolled after hot rolling to produce a much better surface finish and control the strength and ductility through work hardening. Again, four-high mills are used along with lubricants and, if a bright surface finish is required, polished rolls. Depending on the amount of reduction and the final strength required, intermediate anneals are conducted during the cold rolling process. Normally, reductions in the range of 45 to 85% are taken between anneals [9].

### 2.1.1.2 Extrusion

Both cold and hot extrusion methods are used to produce extruded aluminium shapes. Cold or impact extrusions are made by a single sharp blow of a punch into a die cavity that contains a blank or slug of the correct size and shape. Almost all aluminium alloys can be formed by impact extrusion. The slugs are annealed and then generally impact extruded at room temperature, although the temperature may rise to as much as 230°C (450 F) due to the extensive plastic deformation. The slugs are also lubricated prior to extrusion to prevent excessive galling and die wear. Direct hot extrusion is used to make structural shapes. In the direct extrusion process, the cylindrical ingot is preheated and then extruded in the temperature range of 340°C to 510°C (650 to 950 F), depending on the specific alloy. The preheated ingot is placed in a hydraulic press and squeezed at high pressure through a steel die to produce the desired shape. During extrusion, the metal flows most rapidly at the centre of the ingot. Since oxide and surface defects are left in the last 10 to 15% of the ingot, this part of the ingot (called the butt) is discarded. The 6xxxseries alloys, because of their easy extrudability, are the most popular alloys for producing shapes. The 2xxx- and 7xxx-series alloys are used in applications requiring higher strength; however, these alloys are more difficult to extrude [9].

### 2.1.2 Composition of Al-7046 Alloy

Chemical composition of Al-7046 alloy is as following;

- Aluminium, Al 89-92.3%
- Chromium, Cr  $\leq 0.20\%$
- Copper, Cu  $\leq 0.25\%$
- Iron, Fe  $\leq 0.40\%$
- Magnesium, Mg 1.0-1.6%
- Manganese, Mn  $\leq 0.30\%$
- Other, each  $\leq 0.05\%$
- Other, total  $\leq 0.15\%$
- Silicon, Si  $\leq 0.20\%$
- Titanium, Ti  $\leq 0.06\%$
- Zinc, Zn 6.6-7.6%
- Zirconium, Zr 0.10-0.18%

Physical properties;

- Density  $2.82 \text{ (kg/m}^3 \text{ ) } \times 10^3$

## **2.2 Production of Aluminium Foam**

### **2.2.1 Foaming Melts by Gas Injection**

Pure liquid metals cannot easily be caused to foam by bubble a gas into them. Drainage of liquid down the walls of the bubbles usually occurs too quickly to create a foam that remains stable long enough to solidify. However, 10–30% of small, insoluble, or slowly dissolving particles, such as aluminium oxide or silicon carbide, raising the viscosity of the aluminium-melt and impede drainage in the bubble membrane, stabilizing the foam. Gas-injection processes are easiest to implement with aluminium alloys because they have a low density and do not excessively oxidize when the melt is exposed to air or other gases containing oxygen [4].

A variety of gases can be used to create bubbles within liquid aluminium. Air is most commonly used but carbon dioxide, oxygen, inert gases, and even water can also be injected into liquid aluminium to create bubbles. Bubbles formed by this process float to the melt surface, drain, and then begin to solidify. The thermal gradient in the foam determines how long the foam remains liquid or semi-solid, and thus the extent of drainage. Low relative density, closed-cell foams can be produced by carefully controlling the gas injection process and the cooling rate of the foam [4].

### **2.2.2 Foaming Melts by Blowing Agents**

Metal alloys can be foamed by mixing into them a foaming agent that releases gas when heated. The widely used foaming agent titanium hydride ( $\text{TiH}_2$ ) begins to decompose into Ti and gaseous  $\text{H}_2$  when heated above about  $465^\circ\text{C}$ . By adding titanium hydride particles to an aluminium-melt, large volumes of hydrogen gas are rapidly produced, creating bubbles that can lead to closed cell foam, provided foam drainage is sufficiently slow, which requires a high melt viscosity [4].

### **2.2.3 Gas-releasing particle decomposition in semi-solids**

Foaming agents can be introduced into metals in the solid state by mixing and consolidating powders. Titanium hydride, a widely used foaming agent, begins to decompose at about  $465^\circ\text{C}$ , which is well below the melting point of pure aluminium  $660^\circ\text{C}$  and of its alloys. This raises the possibility of creating foam by dispersing the foaming agent in solid aluminium using powder metallurgy processes and then raising the temperature sufficiently to cause gas release and partial or full melting of the metal, allowing bubble growth. Cooling then stabilizes the foam. It begins by combining particles of a foaming agent (typically titanium hydride) with an aluminium alloy powder. After the ingredients are thoroughly mixed, the powder is cold compacted and then extruded into a bar or plate of near theoretical density. This ‘precursor’ material is chopped into small pieces, placed inside a sealed split mould, and heated to a little above the solidus temperature of the alloy. The titanium hydride then decomposes, creating voids with a high internal pressure. These expand by semi-solid flow and the aluminium swells, creating foam that fills the mould [4].

## 2.2.4 Casting using a polymer or wax precursor as template

An open-cell polymer foam mould template with the desired cell size and relative density is first selected. This can be coated with a mould casting (ceramic powder) slurry which is then dried and embedded in casting sand. The mould is then baked both to harden the casting material and to decompose (and evaporate) the polymer template, leaving behind a negative image of the foam. This mould is subsequently filled with a metal alloy and allowed to cool. The use of a moderate pressure during melt infiltration can overcome the resistance to flow of some liquid alloys. After directional solidification and cooling, the mould materials are removed leaving behind the metal equivalent of the original polymer foam. The method gives open-cell foams with pore sizes of 1–5 mm and relative densities as low as 0.05. The process can be used to manufacture foams from almost any metal that can be investment cast. In a variant of the process, the precursor structure is assembled from injection-moulded polymeric or wax lattices. The lattice structure is coated with casting slurry and fired, burning it out and leaving a negative image mould [4].

## 2.3 Production of AFS composite

Various technologies have been proposed for making sandwich panels combining aluminium foam and metallic sheets. The most obvious approach is to start from a sheet of metal foam and to bond face sheets to it, e.g. by adhesive bonding, brazing or diffusion bonding. We shall call this ex-situ bonding. One way is to combine metal foam manufacture with bonding, i.e. to create the bonding in-situ. As metal foams are liquid when they are made, this involves high-temperature processing steps [8]. They are mainly, expanding foam between two sheets, foaming of three-layer composite and structural integral foams.

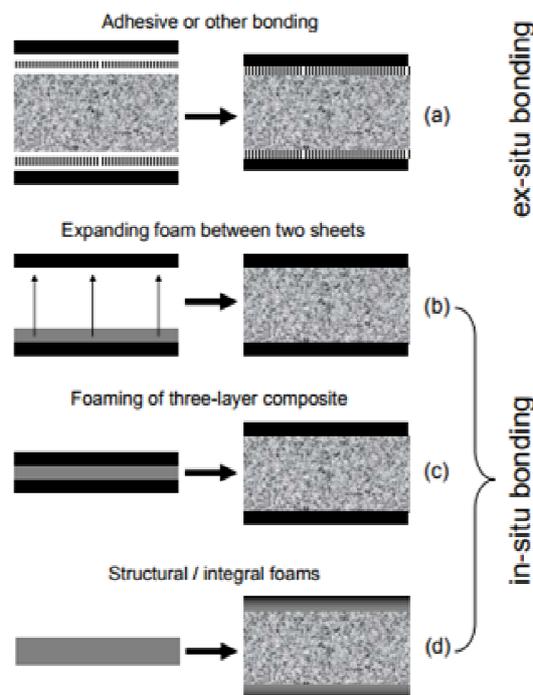


Figure 5: Various technologies for manufacture of AFS [8]

### 2.3.1 Ex-Situ

A foam panel either sliced off a larger block or foamed as a flat product in a mould is bonded to two dense sheets. The properties of the resulting sandwich panel are then given by the interplay of foam, sheet and adhesive and, depending on the parameters chosen, a variety of properties and failure modes are observed [4].

#### 2.3.1.1 Adhesive Bonding

The selection of an adhesive for a given situation is necessary. There is a need for selecting the appropriate adhesive because adhesives are versatile and there is frequent overlap of their usage. It requires detailed knowledge of the properties of a particular adhesive and the application in which it is being used. The adhesive selection process is difficult because there is no universal adhesive that works in every application, and the selection of the proper adhesive is often complicated by the variety of available options. However, adhesive selection includes many factors, such as type and nature of substrates to be bonded, cure and adhesive application method, and the expected environments and stresses that the joint will face in service. Also, the cost of the adhesive may sometimes be an important factor of adhesive selection in a particular production situation. Mechanical actions like impact loads, quasi-static loads, and low and high cycle fatigue loads need to be identified. The mechanical characteristics of an adhesive vary with factors such as high and low strength, brittle and flexible behaviour, as shown in Figure 6 [10].

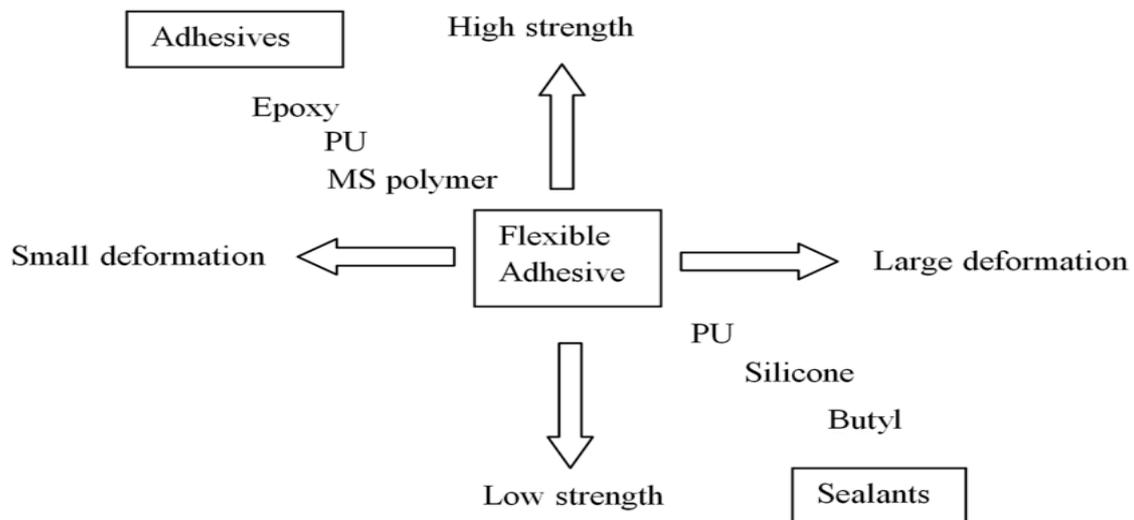


Figure 6: Mechanical characteristics of adhesives [10]

To meet the requirement of a particular application, an adhesive may need one or more of the following properties [10]:

- Strength (shear, peel)
- Resistance to various fluids and chemicals
- Ability to wet the surfaces to be bonded
- Toughness
- Resistance to environmental degradation (including heat and moisture)
- Creep
- Fatigue

The increased use of high-temperature resin-matrix systems for composite materials has necessitated the development of compatible and equally heat-stable adhesive systems. Epoxy adhesives that are frequently used for the composite matrixes, are commonly used to bond composites based on epoxy matrix because of the compatibility between resin and adhesive. Apart from epoxy adhesives, there are several other types of adhesive, as mentioned in Table 5, that are frequently used in various structural applications [10].

Table 5: Typical Properties of Adhesives [10]

<b>Adhesive Type</b>	<b>Typical Adhesive</b>	
	<b>Properties/Applications</b>	<b>Service Temp (°C)</b>
Epoxy	High strength and temperature resistance, relatively low cure temperatures, easy to use, low cost	-40 to +100
Cyanoacrylates	Fast bonding capability to plastic and rubber but poor resistance to moisture and temperature	-30 to +80
Anaerobics	Designed for fastening and sealing applications in which a tight seal is formed without light, heat, or oxygen; suitable for bonding cylindrical shapes	-55 to +150
Acrylics	Versatile adhesives with capabilities of fast curing; tolerates dirtier and less prepared surfaces	-40 to +120
Polyurethanes	Good flexibility at low temperatures and resistant to fatigue, impact resistance, and durability	-200 to +80
Silicones	Excellent sealant for low stress applications; high degree of flexibility and very high temperature resistance; capability to seal or bond materials of various natures, long cure times, and low strength	-60 to +300
Phenolics	Good strength retention for short periods of time; limited resistance to thermal shocks	-40 to +175
Polyimides	Thermal stability, dependent on a number of factors, difficult process ability	-40 to +250
Bismaleimides	Very rigid, low peel properties	-50 to +200

In composite adhesive joints, according to the standard ASTM D5573, there are seven typical characterized modes of failure: adhesive failure, cohesive failure, thin-layer cohesive failure, fibre-tear failure, light-fibre-tear failure, stock-break failure, or mixed failure. In many situations adhesive failure is the limiting factor, although in some applications, for instance in the bonding of thin metal sheet or with composite adherends, adherend failure may be a concern. Interface failure is generally a result of poor surface preparation or incomplete cure. With a true interface failure, the adherend is exposed at the fracture surface [10].

In the manufacture of automobiles the adhesives joints are almost always used as basic sealant materials or in noncritical secondary structures. In the manufacture of aircraft the use of adhesively bonded joints has also largely been limited to secondary noncritical structures such as aerodynamic fairings and wing panels. Therefore, the use of adhesives in truly structural applications has been quite limited. The reasons for these limitations are twofold: First one a concern about the fatigue and durability behaviour of bonded structural components over the expected lifetime of the vehicle and second one the fracture behaviour of adhesive bonded joints, particularly those with dissimilar adherends (i.e., composite to metal), is still not well understood, since the adhesive joints must perform satisfactorily under service conditions, which include static and dynamic loadings and exposure to hostile environments such as water, petrol, other organic solvents, in many instances, combinations of these conditions may be experienced [10].

Current applicable conventional design rules and standards for joining primary composite structures usually lead to local thickening of the fastener area and to a large number of fasteners distributed over several lines to prevent or delay the different failure modes at the interface. The certification approach follows a strict fail-safe philosophy with the sizing strategy that the additional fasteners are capable of carrying full limit load. Therefore, compared to their counterparts in metallic structures, the interfaces in composite thin-walled structures induce a significant weight and cost penalty and mitigate the technical and economic benefits expected from the massive introduction of composites in aero-structures [11].

The joining of thin-walled composite structures by adhesive bonding, without using fasteners, is a promising method to reduce aircraft cost and weight. Adhesive bonding also brings several technical advantages such as the avoidance of detrimental stress concentrations and the avoidance of fibre cuts associated with the drilling of holes for introducing fasteners. For secondary structures, adhesive bonding is common practice but until today the certification rules, that are applicable for primary bonded structures, prevent the use of boltless bonded joints for primary structures, as a result of earlier experiences, where the interpretation of the rules led to in-service premature failure incidents on adhesively bonded joints [11].

The use of bonded joints in primary structures is also prevented by the inability of the existing non-destructive testing techniques to capture all types of defects in the bond-line, the requirement to redesign aircraft parts such as the load to be transferred by pure shear of the bond-line, the susceptibility of the joint (adhesive and adhesive/ substrate interface) into environmental ageing and pre-bond contamination as well as the unknown fatigue behaviour of bonded joints [11].

Despite the many advantages of adhesive bonding, its use is still limited due to low durability of the joint when the structure is exposed to hostile environmental conditions. The effect of moisture on the strength of adhesively bonded joints is significant due to the deterioration of the adhesive layer and the interface. The strength of joint decreases dramatically when aged in hot/wet environments, as has been demonstrated in composite bonded joints [10].

The major limitations of adhesive bonding are summarized as follows [10]:

- There is no universal adhesive. Selection of the proper adhesive is often complicated by the wide variety of available options
- The joint cannot be disassembled readily. Assembly times may be greater than for alternative methods, depending on the curing mechanism. Elevated temperatures may be required, as well as specialized fixtures
- Some adhesives contain objectionable chemicals or solvents, or produce them on curing
- Surface preparation and cleanliness, adhesive preparation, and curing can be critical if good and consistent results are to be obtained
- Some adhesives are quite sensitive to the presence of grease, oil, or moisture on the adherend surfaces to be joined. Surface roughness and wetting characteristics must be controlled
- The thermal residual stresses can be induced
- High-strength adhesives have poor impact characteristics (often brittle). The toughness of an adhesive joint may decrease considerably under impact loading conditions
- Most industrial adhesives are not stable above 180°C. Oxidation reactions are accelerated, thermoplastics can soften and melt, and thermo-sets decompose. While some adhesives can be used up to 260°C, elevated temperatures are usually a cause for concern
- Many structural adhesives deteriorate under certain operating conditions. The adhesive can be subjected to environmental effects. Therefore, the durability and reliability of a joint over an extended service lifetime may be questioned. Long-term durability and life expectancy are difficult to predict
- The joint design is thickness-limited and only shear loading is acceptable
- It is difficult to determine the quality of an adhesive bonded joint by traditional non-destructive techniques, although some inspection methods have been developed that give good results for certain types of joints [10].

### **2.3.1.2 Diffusion Welding**

Diffusion bonding or diffusion welding is a solid state joining process. This bonding technique is based on the atomic diffusion of elements at the joining interface. The diffusion welding interface has same physical and mechanical properties as base metal. The strength of joining depends on pressure, temperature, time of contact and the cleanness of the interface. Diffusion bonding needs longer time than the other welding processes [12].

Diffusion bonding is a joining process where in the principal mechanism for joint formation is solid state diffusion. The solid phase diffusion bonding of the product is used in various industrial fields to make making to high performance and making to a high function near net shape in addition. Diffusion bonding offers many advantages, mainly the strength of the bonding line, which is equal to the base metals. The microstructure at the bonded region is exactly the same as the parent metals. On the other hand, this advantage joining process

requires several strictly controlled conditions; clean and smooth contacting surfaces which are free from oxides, etc., high temperature condition to promote diffusion process [12].

In diffusion bonding, the bond strength is achieved by pressure, temperature, time of contact, cleanness of the surfaces. The strength of the bond is primarily due to diffusion rather than any plastic deformation. Diffusion bonding is an attractive manufacturing option for joining dissimilar metals and for making the component with critical property continuity requirements. Unlike other joining processes the diffusion bonding process preserves the base metal microstructure at the interface. More importantly no localized thermal gradient is present to induce distortion or to create residual stresses in the component [12].

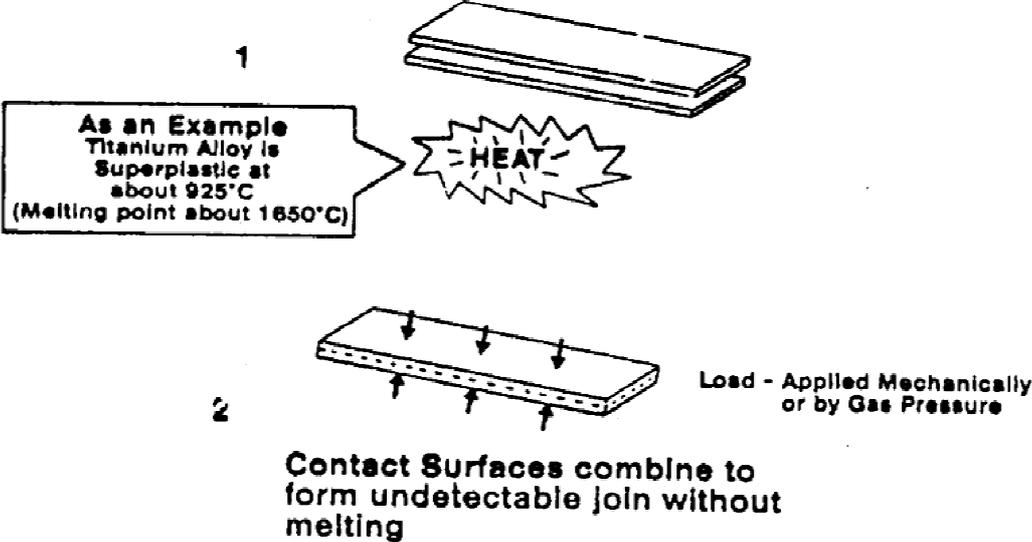


Figure 7: The Basic Processes of Diffusion Welding [12]

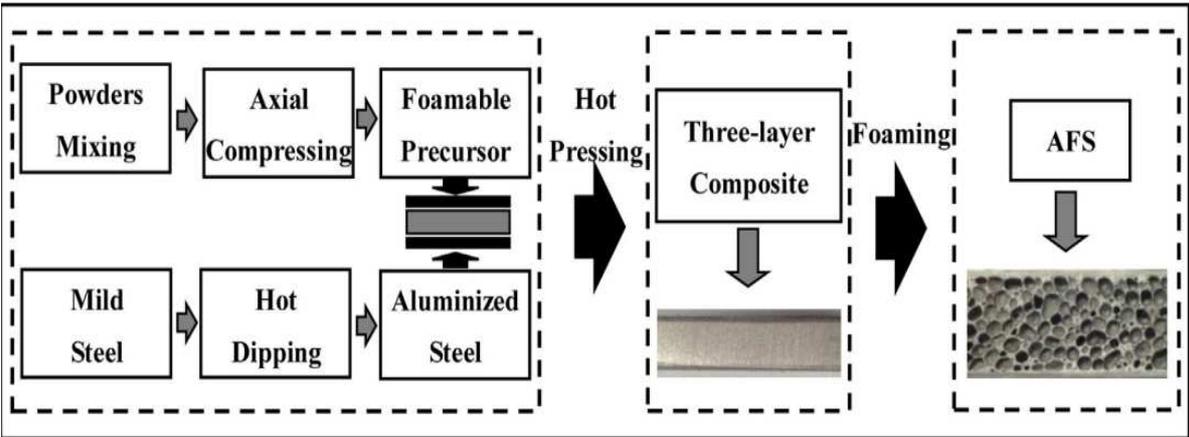


Figure 8: Schematic illustration of process of fabricating AFS panels by diffusion welding method [13]

### 2.3.1.3 Brazing Bonding

Brazing and soldering require the application of a number of scientific and engineering skills to produce joints of satisfactory quality and reliability. Brazing employs higher temperatures than soldering, but the fundamental concepts are similar, particularly with respect to metallurgy and surface chemistry (Table 6). However, joint design, materials to be joined, filler metal and flux selection, heating methods, and joint preparation can vary widely between the two processes. Economic considerations involving filler metal and process technology are also varied, particularly in relation to automated techniques and inspection and testing. Brazing and soldering are performed in many industries, from exotic applications in the electronics and aerospace field to everyday plumbing applications [14].

Table 6: Comparison of soldering, brazing, and welding [14]

Parameter	Process		
	Soldering	Brazing	Welding
Joint formed	Mechanical	Metallurgical	Metallurgical
Filler metal melt temperature, °C (°F)	<450 (<840)	<450 (<840)	<450 (<840)
Base metal	Does not melt	Does not melt	...
Fluxes used to protect and to assist in wetting of base-metal surfaces	Required	Optional	Optional
Typical heat sources	Soldering iron; ultrasonics; resistance; oven	Furnace; chemical reaction; induction; torch; infrared	Plasma; electron beam; tungsten and submerged arc; resistance; laser
Tendency to warp or burn	Atypical	Atypical	Potential distortion and warpage of base-metal likely
Residual stresses	...	...	Likely around weld area

Brazing is a process for joining solid metals in close proximity by introducing a liquid metal that melts above 450 °C (840 °F). A sound brazed joint generally results when an appropriate filler alloy is selected, the parent metal surfaces are clean and remain clean during heating to the flow temperature of the brazing alloy, and a suitable joint design is used. Like the other joining processes, brazing encompasses a variety of scientific disciplines (for example, mechanics, physics, and chemistry). Recently, the demands of more-sophisticated structures have forced technicians and engineers to encourage metal producers to apply their metallurgical knowledge to produce brazing filler metals that meet more-specific needs. To ensure the production of good brazed joints, the technicians and engineers also had to appeal to mechanical engineers for improved joint design, to chemical engineers for solutions to corrosion problems, and to metallurgists and ceramists for proper material selection [14].

Brazing proceeds through four distinct steps [14];

- The assembly or the region of the parts to be joined is heated to a temperature of at least 450 °C (840 °F)
- The assembled parts and brazing filler metal reach a temperature high enough to melt the filler metal but not the parts
- The molten filler metal, held in the joint by surface tension, spreads into the joint and wets the base metal surfaces
- The parts are cooled to solidity, or "freeze," the filler metal, which is held in the joint by capillary attraction and anchors the parts together by metallurgical reaction and atomic bonding [14].

Strong, uniform, leak-proof joints can be made rapidly, inexpensively, and even simultaneously. Joints that are inaccessible and parts that may not be joinable at all by other methods often can be joined by brazing. Complicated assemblies comprising thick and thin sections, odd shapes, and differing wrought and cast alloys can be turned into integral components by a single trip through a brazing furnace or a dip pot. Metal as thin as 0.01 mm (0.0004 in.) and as thick as 150 mm (6 in.) can be brazed [14].

Brazed joint strength is high. The nature of the inter-atomic (metallic) bond is such that even a simple joint, when properly designed and made, will have strength equal to or greater than that of the as-brazed parent metal. Complex shapes with greatly varied sections can be brazed with little distortion, and precise joining is comparatively simple. Unlike welding, in which the application of intense heat to small areas acts to move the parts out of alignment and introduces residual stresses, brazing involves fairly even heating and thus part alignment is easier [14].

Advantages of brazing, including the following [14];

- Economical fabrication of complex and multi-component assemblies
- Simple method to obtain extensive joint area or joint length
- Joint temperature capability approaching that of base metal
- Excellent stress distribution and heat-transfer properties
- Ability to preserve protective metal coating or cladding
- Ability to join cast materials to wrought metals
- Ability to join non-metals to metals
- Ability to join metal thicknesses that vary widely in size
- Ability to join dissimilar metals
- Ability to join porous metal components
- Ability to fabricate large assemblies in a stress-free condition
- Ability to preserve special metallurgical characteristics of metals
- Ability to join fibre and dispersion strengthened composites
- Capability for precision production tolerance
- Reproducible and reliable quality control techniques [14]

Several elements of the brazing process must be understood in order to produce satisfactory brazed joints [14];

- Filler metal flow
- Base metal characteristics
- Filler metal characteristics
- Surface preparation
- Joint design and clearance
- Temperature and time
- Rate and source of heating [14]

Brazing has been embraced by the engineering community and has now reached a very successful plateau within the joining field. This has come about because of the [14]:

- Development of new types of brazing filler metals (rapid solidification amorphous foils and titanium-added filler metals for ceramic joining)
- Availability of new forms and shapes of filler metals
- Introduction of automation that has brought brazing processes to the forefront in high-production situations
- Increased use of furnace brazing in a vacuum, as well as active and inert-gas atmospheres [14]

Aluminium is a commonly used base material for brazing. It is easily fabricated by most manufacturing methods, such as machining, forming, and stamping. Although most brazing processes are applicable, the use of aluminium requires more attention. It is different from most metals in two ways: it quickly reforms its tenacious oxide coating whenever a reactive form of oxygen is present, such as air or water, and it is always brazed at temperatures close to its melting point [14].

Table 7; Melting ranges and brazeability of some common aluminium alloys [14]

ALLOY	MELTING RANGE		BRAZEABILITY
	°C	°F	
NON-HEAT-TREATABLE WROUGHT ALLOYS			
1350	646-657	1195-1215	A
1100	643-657	1190-1215	A
3003	643-654	1190-1210	A
3004	629-652	1165-1205	B
5005	632-652	1170-1205	B
5050	627-652	1160-1205	B
5052	593-649	1100-1200	C
HEAT-TREATABLE WROUGHT ALLOYS			
6053	593-652	1100-1205	A
6061	593-649	1100-1200	A
6063	616-654	1140-1210	A
6951	616-654	1140-1210	A
7005	607-649	1125-1200	B

The commonly brazed heat-treatable wrought alloys are the 6xxx series. Because the 2xxx and 7xxx series of aluminium alloys have melting points that are too low, they are not normally brazeable. Exceptions are the 7072 alloy, which is used for cladding material only, and 7005 alloy. Alloys with a solidus temperature above 595 °C (1100 °F) are readily brazed using the aluminium-silicon filler metals. Filler metal alloys used in flux brazing usually contain between 7 and 12% silicon balanced aluminium, and tramp metals are controlled to levels below 0.2%. Alloys employed in fluxless brazing use higher percentages of silicon (>9%) and have varying additions of magnesium to enhance oxide film modification to promote wetting, as well as reduce the partial pressures of oxygen bearing gases in the chamber. These alloys are primarily found in clad form [14].

**Fluxes;** brazing that is performed in air or other oxygen containing atmospheres requires the use of a chemical flux. These fluxes, whether used in torch, furnace, or dip brazing, or in braze pastes, usually contain chlorides and/or fluorides, the fumes of which are hazardous to the operator if inhaled. Flux fumes must be exhausted, not only for health reasons, but because they are highly corrosive to most metals. Fluxes act as deoxidizers and prevent re-oxidation by coating the surfaces [14].

They are used to aid the wetting and flow of the filler metal on deoxidized and clean aluminium. There are four generic types of fluxes in two basic formulas: chloride active and fluoride active. Chloride-active fluxes consist of either a straight chloride flux or a chloride flux modified with zinc. Fluoride-active fluxes consist of either a straight fluoride or a modified fluoride that produces an inert film over the brazed parts after brazing. In both cases, the modification is intended to reduce corrosion in post brazed products. In torch brazing, fluxes can be applied dry or mixed with either alcohol or water and painted, or sprayed, or they can be applied by dipping the joining parts [14].

A suitable flux for torch or furnace brazing must [14];

- Begin to melt at a temperature low enough to minimize oxidation of the parts
- Be essentially molten at the time the filler metal melts
- Flow over the joint and filler metal to shield them from oxidizing gases
- Penetrate oxide films
- Lower the surface tension to encourage wetting
- Remain liquid until the filler metal has melted
- Be relatively easy to remove after brazing is complete [14]

For heat-treatable alloys, chemical cleaning is necessary to reduce the tenacious oxide films, which tend to be thicker, because of thermal processing methods such as solution heat treatment. Aluminium-silicon alloys often require a special nitric-hydrofluoric acid mix to remove the excess silicon smut that is not removed by alkaline (5% NaOH) or nitric-acid solutions alone. If necessary, stainless steel wire brushes, steel wool, or abrasive cloth can be used to achieve abrasive cleaning. Thin, high-silicon-content braze foil should only be degreased to prevent the loss of available filler metal that is needed at the braze joint [14].

There are mainly 4 different types of brazing as following;

**Dip Brazing;** is done by immersing the brazing assembly and fixture into a molten bath of flux. The flux acts as both a heating mechanism to bring the parts to temperature, as well as a deoxidizer. Prior to immersion, the part and fixture must be preheated to remove moisture and prevent solidification of the flux during immersion. Preheating is done in a hot-air furnace at approximately 540 °C (1000 °F) for about 0.5 h. Duration of the preheat cycle varies with the complexity of the brazement, its size, and the size of the fixture. Nevertheless, even with small waveguide components and thin box brazements that require a decreased preheat time, using a preheat cycle time of 0.5 h is still recommended. Moisture removal is necessary to prevent steam explosions when the part is lowered into the bath. Steam explosions can be very hazardous to the operator [14].

Initial temperatures are determined by attaching a thermocouple to the aluminium assembly. Time and temperature readings are taken and used for subsequent parts as a mechanism to control the braze quality. Adequate salt drainage is necessary for several reasons. Salt must be removed to prevent trapping and the possible corrosion of the parts. Adequate drainage is necessary to allow filler metal solidification. Retention of excess salt in the assembly depletes the level of the molten salt in the dip pot, necessitating more-frequent additions of dry flux to the bath and resulting in higher costs. The bulk of the salt is removed by immersing the fixture and brazement in a bath of boiling water. Then, the brazement is separated from the fixture and put through a series of water baths to remove more salt. Final baths, which use distilled water, are changed at intervals until chemical tests show an acceptable limit of salt in the clear runoff water [14].

Concurrently, the fixtures are further boiled and then blasted with water to remove any residual salt. The molten flux bath is regularly dehydrated by immersing clean sheets of 1100 or 3003 series aluminium to remove water, oxides, and other contaminants for an amount of time that varies from bath to bath. The sheets must be cleaned and dried to maintain their effectiveness. They can be used repeatedly [14].

**Furnace Brazing;** Standard fluxes must be used in furnace brazing because of the oxide atmospheres. Flux can be applied by brushing, dipping, or spraying. Furnace brazing is used for large loads that require good temperature control. Stop-offs is often used on parts and fixtures to inhibit the flow of molten filler metal to certain areas and to prevent brazing to the fixtures. Special fluxes can be used with special furnaces that have reduced-oxygen atmospheres. One such flux is NOCOLOK, which has modest cleaning and fluxing ability and requires well-cleaned parts. During the brazing process, the chamber is filled with flowing dry nitrogen. At the completion of brazing, the parts are coated with an inert residue from the flux. The parts do not require post-braze cleaning to remove the flux residue. The temperature accuracy requirement for these aluminium brazing furnaces is usually  $\pm 5.5$  °C ( $\pm 10$  F). However,  $\pm 3$  °C ( $\pm 5$  F) is preferred, especially where critical components are being brazed [14].

**Torch Brazing;** is used in either manual or automatic fabrication, as well as in repair operations. Torch brazing ranges from simple tube-to-tube joints to more-complex and mechanized assemblies. Torch brazing is similar to oxy-fuel gas welding in that the heat to effect the joint is applied locally. Oxyacetylene, oxy-hydrogen, and oxy-natural gas are used in commercial torch brazing applications. Because aluminium shows no change in colour when hot, it is difficult to know when parts approach the brazing temperature. Fluxes that melt at a slightly lower temperature than the filler metal can serve as a temperature indicator. Joint areas are pointed with slurry of brazing flux. A soft, slightly reducing flame is played over the entire area. Filler metal can be either preplaced or force fed. After brazing, residual flux chloride must be removed [14].

**Flux-less Vacuum Brazing;** The main advantage of flux-less vacuum brazing is that no flux removal is necessary. If assemblies have been made using non-heat-treatable alloys, then they are ready for the next operation. If heat-treatable alloys are brazed, then a subsequent heat treatment will be necessary to reach the strengths required. The required equipment is a vacuum furnace that can reach a vacuum of  $133 \times 10^{-5}$  Pa ( $10^{-5}$  torr) or lower, is leak tight, and is able to obtain temperature accuracy of  $\pm 3$  °C ( $\pm 5$  °F) on the load being brazed. Flux-less brazing is ideal for braze sheets where no flux is required and assemblies can easily be stacked and fixture [14].

Magnesium in some form is usually required to expedite the break-up of surface oxides and to facilitate the wetting mechanism. Magnesium is supplied by several techniques. It can be an integral part of the filler metal or base metal or it can be a part of other alloys used in the assemblies, such as the 5xxx series of aluminium-magnesium alloys. It can also be supplied in the form of chips or pre-alloyed aluminium-magnesium ingots [14].

Vacuum furnaces must be cleaned periodically to remove magnesium and magnesium oxide that condenses on the cold walls of the furnace and other appendages to those walls. These residues may, in time, short some of the electrodes and tend to be hygroscopic to water. Water is a contaminant that is very difficult to remove in vacuum [14].

### **Post-braze Heat Treatment**

Dip-brazed heat-treatable assemblies can be spray quenched or immersion quenched after most of the salt has been drained and while the parts still remain at about 480 °C (900 °F). This puts the parts into a solution-heat-treated stage for subsequent precipitation hardening. Heat-treatable assemblies usually are in the annealed or soft condition after furnace, torch, or flux-less brazing. Clean assemblies are usually heated at temperatures ranging from 480 to 515 °C (900 to 960 °F) for a period of time that allows the magnesium silicates to go into solid solution prior to quenching. The quenched parts are then aged for a period of time that allows precipitation hardening to take effect. The amount of time depends on the alloy system and on whether the parts are made from wrought or cast alloys. A typical precipitation heat treatment for 6061 shapes and tube is 160 °C (320°F) for 16 to 20 h [14].

### 2.3.2 In-Situ

Another way is to combine metal foam manufacture with bonding, i.e. to create the bonding in-situ. As metal foams are liquid when they are made, this involves high-temperature processing steps [8].

Face sheet and foam core have to be made of alloys with different melting points since foaming takes place in the semi-solid or even liquid state. At these temperatures the face sheet must not melt. In early foaming practice furnaces were heated to temperatures well above the melting point of the foam able material (e.g. 750°C) to ensure a rapid temperature increase in the AFS precursor material after it had been placed inside the furnace. The corresponding temperature course is shown in Figure 9 [8].

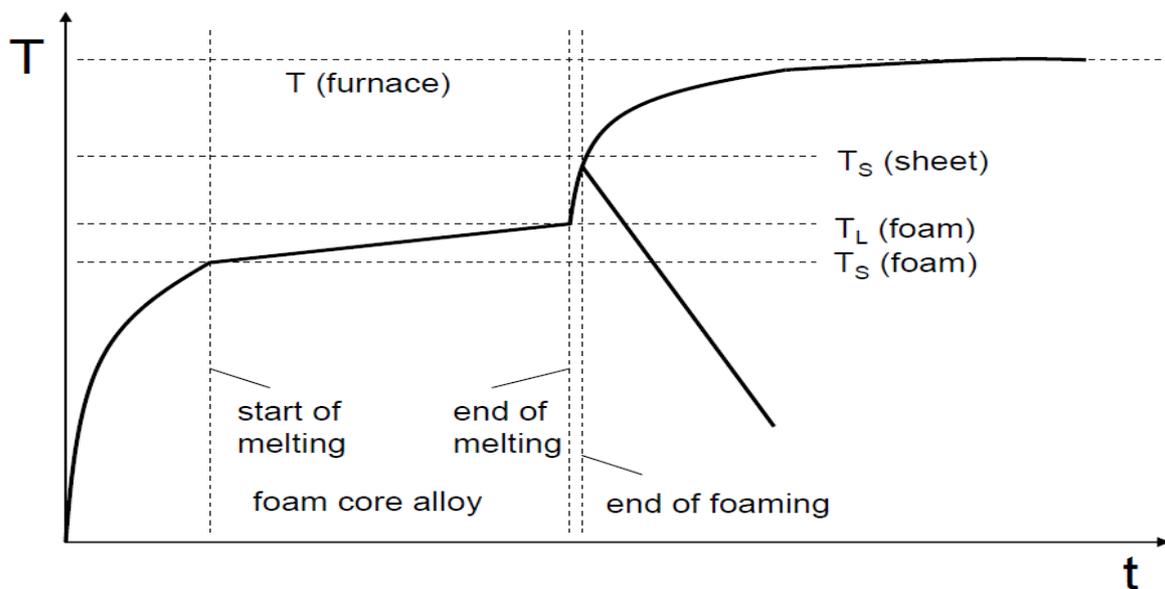


Figure 9: Temperature course of AFS foaming in furnace kept at a constant temperature  $T > T_L$  [8]

As face sheets and foam able core are in close contact and the heat conductivity of the two alloys is high, temperature is approximately the same throughout the material. The material with the lower melting temperature (the foam) therefore limits the increase in temperature during melting and therefore effectively cools the face sheets as long as it is still semi-solid. After complete melting of the foam, temperature rises rapidly and the face sheets will also melt unless the sample is taken out of the furnace for cooling [8].

The solidification temperature  $T_S$  of the face sheets therefore has to be above the liquidification temperature  $T_L$  of the foam. This restricts the number of usable aluminium alloy combinations. Early choices of materials were pure aluminium or 3003 alloy ( $AlMn_1$ ) for the face sheets and near-eutectic Al-Si alloys for the foam. No problems with melting are encountered when using steel or titanium face sheets, but in these cases inter-metallic compounds can be formed with the possible danger of embrittlement [8].

The situation is more favourable when the heating process is actively controlled as it is in modern industrial practice. Here, the foam-able material is heated up to an end temperature as quick as possible, after which the temperature is kept approximately constant. This offers the possibility to create the foam just above the solidification temperature of the foam and chose face sheets from alloys which melt just above this temperature, see Figure 10 [8].

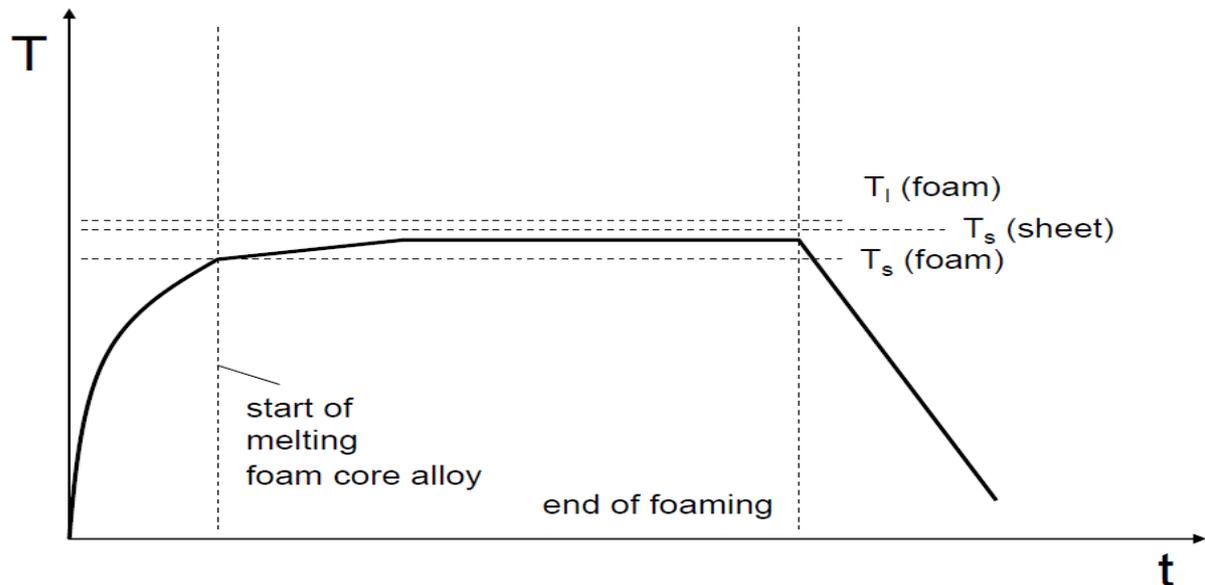


Figure 10: Temperature course of AFS foaming in furnace with a dynamic heating profile ensuring that  $T < T_s$  [8]

In this way a wider range of alloy combinations can be processed. Three groups of aluminium alloy face sheets are currently being used [8]:

- Non heat treatable 3000 alloys, mainly 3103 ( $AlMn_1$ )
- Non heat treatable 5000 alloys: 5005 ( $AlMg_1$ ), 5754 ( $AlMg_3$ ) or 5083 ( $AlMg_{4.5}$ )
- Heat treatable 6000 alloys: 6016, 6060 or 6082 (Al-Mg-Si system) [8]

For the foam core, two groups of alloys based on the Al-Si system have been developed and tested. Currently, the alloy  $AlSi_6Cu_x$  ( $x \approx 3...7$ ) is preferred for its low solidification temperature and very good foaming behaviour. In all cases the foam-able core contains  $TiH_2$  in the usual contents [8].

### 2.3.2.1 Three Layer Composite

A three-layer composite comprising a foam able (containing  $\text{TiH}_2$  as a blowing agent) aluminium alloy sheet as a core layer and two face sheets on both sides (usually aluminium alloy, but steel and titanium have also been used) is made by extrusion or powder rolling and various subsequent rolling operations, after which the core layer of the panel is expanded by heating to the foaming temperature. The core expands, while the face sheets remain both solid and firmly bonded to the foam during this step. The advantages of this process are obvious: a pure metallic bonding is maintained that is less heat sensitive than an adhesive and can be used to manufacture heat resistant non-inflammable structures. In addition, recycling of such AFS should be easier than recycling of polymer-bonded sandwich panels. By shaping the three-layer precursor prior to foaming, a 3D shape can be produced, see Figure 11, which is hardly possible by ex-situ bonding techniques. Disadvantages include that the number alloy combinations are restricted and high-temperature tooling is required [8].

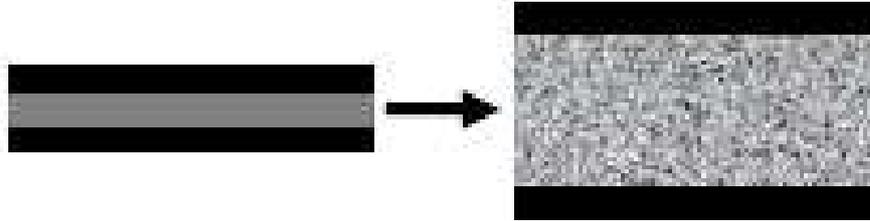


Figure 11: Foaming of three-layer composite [8]

### 2.3.2.2 Expanding Foam between Two Sheets

Since preparing a three-layer composite is an additional process step one is tempted to try making AFS by merely expanding a piece of foam able precursor between two face sheets kept at distance Figure 12. This procedure, however, leads to difficulties since the oxide layers on both the face sheets and the foam make a true metallic bonding difficult. Moreover, temperature control is more difficult in this case since heat has to be introduced into the foam through the face sheets and the danger of melting these is high in case of aluminium face sheets. Therefore, this technology is still in an experimental stage [8].

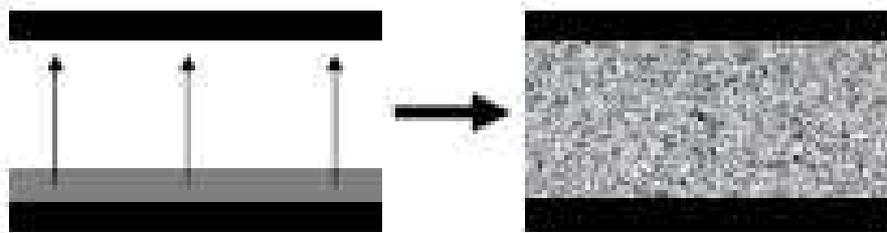


Figure 12: Expanding foam between two sheets [8]

### 2.3.2.3 Structural / Integral Foams

Yet another strategy to manufacture AFS is to manipulate the foaming process in a way that a dense skin is created during foaming without using any dense sheets, Figure 13. By expanding the precursors used for the processes described above under special conditions can lead to the metal equivalent of structural foam in polymer foam technology, i.e. a foamed core with a thick, nearly dense skin which originates from the material used for foaming, see Figure 14. Recently a new technology has been developed which allows for making structural aluminium or magnesium alloy foam parts in a die casting machine by injecting liquid alloy and blowing agent into the die and applying an appropriate pressure profile. In this way flat panels can be produced with a near-dense outer skin [8].

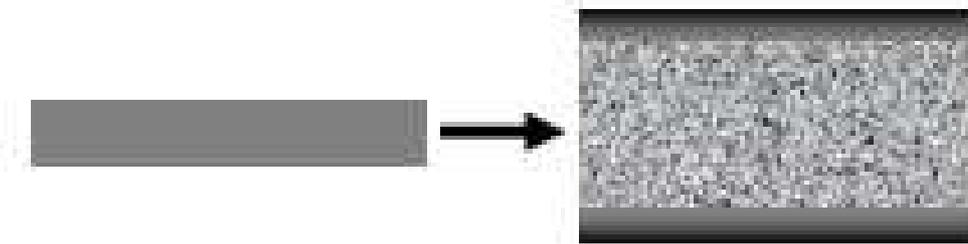


Figure 13: Structural / integral foams [8]

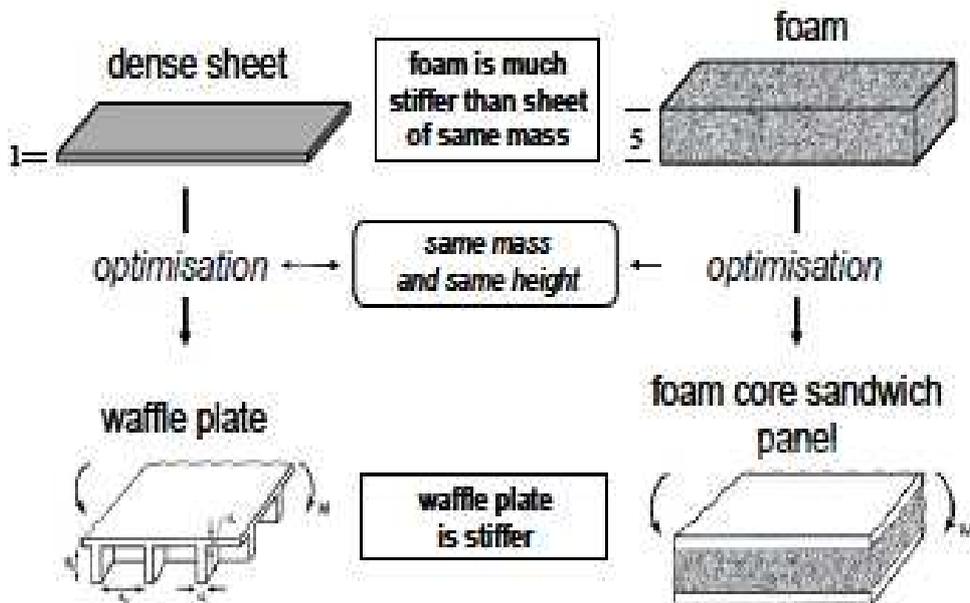


Figure 14: Optimisation of dense sheets and foams [8]

## 2.4 Decision of Brazing Bonding for AFS Production

The AFS composites I produced from Al-7046 should obtain good level of strength, shock absorption, elastic modulus and thermal expansion which should be consistent with the high temperature because mostly the applications of my AFS components are for aerospace and automobile sectors. If you consider the aerospace applications, temperature range is very high thus it is necessary to have good thermal stability of the AFS composite. Another important point is also for automobile sectors that the applications of AFS composites on the floor panel for the trucks, body in white part of the vehicles. Also having good thermal stability of the composites is a plus.

The most obvious and straightforward approach, adhesive bonding, is conducted by combining of the pre-fabricated foam core with cover sheets directly using polymeric adhesive. Associated with the adhesive, this approach has certain problems of low strength, additional mass, difficult recycling, not feasible at high temperature [13], reduced stiffness and high costs. For these reasons, this method is not useful for producing AFS composites.

Diffusion bonding, as a type of simple joining, demonstrates its potential in the joining of similar and dissimilar metals, alloys, non-metals. Conventional diffusion bonding of steel and Al alloy involved a high vacuum system to maintain clean surfaces. It put a limit on the size of the component and required complicated equipments to improve reliability [13]. Dimension limitation of the components for hot pressing and other processes which explained previously do not fit with my AFS composite and this method is difficult to produce my desired AFS composite in terms of cost of the equipments for diffusion bonding and monitoring vacuum systems.

Before starting to my thesis work, I already had Al-7046 plates and Al-foam thus my research was about which ex-situ joining method fits the best way to join them together.

However, there are also in-situ methods to produce AFS composite directly from the base material such as three layers composite. The negative side of this method is number alloy combinations are restricted and high-temperature tooling is required. There is also another method so called expanding foam between two sheets however this method is still in experimental stage because of the controlling the temperature and oxide layer are really difficult. The next one is structural / integral foams which processing by injection liquid alloy and blowing agent into the die and applying an appropriate pressure profile. In this case, high equipment investment and material costs are required.

There is another method that the foam core is metallurgically bonded to the cover sheets. In this process, a foam-able precursor and two dense cover sheets on both sides were clad-bonded, and then it was foamed to be AFS panel by heat treatment. This approach had been brought to a level of sophistication to allow for manufacturing AFS panels of satisfactory quality. In recent years, there is a new technology via self-propagating high temperature synthesis (SHS). In this way, the Al cover sheet was bonded to aluminium foam core by the melting interface which was caused by the generated heat from the SHS exothermic reaction. But this method had a certain limitation. Since the SHS reaction took place in the foam core/cover sheets, the foam porosity should be low to provide enough surface area for occurrence of the SHS reaction [13].

For that reason, the application of this SHS method is both difficult in terms of processing and monitor of the operation, also the cost of the method. An alternative processing route that both the fabrication of the Al–Si–Cu alloy foam-able precursor and welding between the precursor and Al alloy plates were simultaneously achieved by friction stir welding (FSW). However, this method was applicable only to parts with certain shapes (preferably with round cross section) and which also had to be machined to provide conical ends. Respecting the rectangle geometry of my AFS composite, FSW method also does not excellent fit for production [13].

We can implement brazing bonding processes with these features; strong, uniform, leak-proof joints can be made rapidly, inexpensively, and even simultaneously. We can achieve extensive joint length with simple method and suitable to economical fabrication. It is perfectly suit for heat treatable wrought alloys with Al-foams. Another positive point is not going up to high temperatures which makes our processes safer and easier. The easiest and most convenient one was furnace brazing in Argon gas atmosphere for producing AFS composites in terms of economical aspect and difficulty of the implementation. It is in Argon gas atmosphere in order to prevent oxidation on the composite because of the high reactivity of aluminium towards oxygen. As previous experimental papers are done about brazing processes, brazing processes are also proposed: for instance, Al86SiMg alloy was adopted as a filler material for the joining of Al-foam to Al-sheet [15].

## **2.5 Decision of Zinc Base Brazing Material for AFS Production**

It is recommended to use zinc-aluminium group, tin-zinc group, cadmium-zinc group and tin-lead-zinc group as soldering-brazing alloys also recommended to use zinc-aluminium group alloys for 8xxx series of wrought aluminium alloys and tin-zinc group alloys for 7xxx series of wrought aluminium alloys.

Creation of the AFS composite with Al-7046 and Al-foam is decided and Al-7046 alloy has got zinc alloying element around 6.6÷7.6%. The brazing of aluminium is different from brazing of other metals in some ways. The most important different is aluminium oxide forms a more tenacious and refractory oxide which making mandatory the use of corrosive fluxes designed for aluminium. A second difference is that the soldering of aluminium requires special techniques to produce flow into certain types of joints. Thirdly, the solder composition influences corrosion resistance of solder joints much more with aluminium than with copper, brass or steel. With proper attention to details, however, a large variety of solder joints can be made in aluminium base materials. The use of solders with high zinc or tin content is usually recommended. The tin-lead solders are not recommended because the joint has poor resistance to corrosion and exhibits a loss of strength due to aging [16].

Table 8: Typical solders for aluminium [16]

Composition (percent)	Temperature (°F)		Density (lb per cu in.)	Wetting Ability on Aluminium	Flux Type Commonly Used	Relative Corrosion Resistance
	Solidus	Liquidus				
100 Zn	787	787	0.26	Good	Reaction	Very Good
95 Zn-5 Al	720	720	0.24	Good	Reaction	Very Good
91 Sn-9 Zn	390	390	0.26	Fair	Chemical& reaction	Fair
60 Sn-40 Zn	390	645	0.26	Good	Reaction	Good
30 Sn-70Zn	390	708	0.26	Good	Reaction	Good

As shown on the table 8, pure zinc joining alloy is the best application for brazing applications for my case. Wetting ability on aluminium is good and relative corrosion resistance is also very good. Thus, I decided to use pure zinc as brazing joining material firstly. Pure zinc is also easy to obtain with economical prices comparing the other materials.

Also, zinc component helps in preventing galvanic corrosion. Zinc based hard solders (below 400 °C melting point and bulk shear strength approaching 175 MPa or 25,000 psi) use fluxes that require over-heating of the aluminium alloy [17] also the solubility of Zn in Al is the largest among all elements [18].

As second, zinc with some small amount of aluminium is also useful for brazing process as brazing joining material shown in table 8. There is 95%Zn+5%Al in the second place on the table.

However, the most widely used of these is 98%Zn+%2Al, having a melting range of 376-385°C. These alloys have been used at temperatures up to 550°C using all available heat sources [17].

Table 9: Soft and hard aluminium solders [17]

92Sn/8Zn	<b>98Zn/2Al</b>	76Sn/21Zn/3Al
91Sn/9Zn	97Zn/3Al	71.5Sn/25Zn/3.5Al
80Sn/20Zn	95Zn/5Al	67Sn/27Zn/3Al
75Sn/25Zn	90Zn/10Al	55Sn/33Zn/11Al/1Cu
70Sn/30Zn	85Zn/15Al	67Sn/17Zn/15Al/1Cu
60Sn/40Zn	80Zn/20Al	90.3Sn/9.1Zn/0.6Al

On my thesis work, I aimed to obtain AFS components by joining Al-7046 sheets to Al-foams with brazing method in argon gas atmosphere. Firstly pure zinc and then secondly 98%Zn-2%Al fillers were selected as joining materials in the production of AFS composites with brazing method. AFS composites were manufactured with both pure zinc and 98%Zn-2%Al fillers.

### 2.5.1 Microstructure of Al-Zn and Zn-Al Alloys

The solubility of Zn in Al is the largest among all elements, showing a maximum of 67% at 654°K. This is obviously due to the fact that Zn and Al do not form inter-metallic phases or, in other words, the interaction between Al and Zn atoms is fairly weak. At room temperature (298 °K), the solubility of Zn in Al amounts 0.85% and the one of Al in Zn is smaller than 0.5%. The atomic radius of Al is 0.143 nm, while the one of Zn is 0.134 nm, this difference of approximately 7% having a great influence on the microstructure of the Al-Zn and Zn-Al alloys. Al-Zn or Zn-Al alloys can gradually (asymptotically) approach the equilibrium state after, for instance, rapid quenching and a prolonged ageing at room temperature. That process can be accelerated at an elevated temperature several tens K's above room temperature. In such a state, the alloy contains two phases;  $\alpha$ -phase (fcc, the matrix, M) containing  $\approx 99\%$  Al and  $\approx 1\%$ Zn, and  $\beta$ -phase (hexagonal, the precipitates), often denoted in literature as  $\beta$ (Zn), having  $\approx 99.5\%$ Zn and  $\approx 0.5\%$  Al. It is said that the phase  $\alpha$ (M/ $\beta$ ) is in equilibrium with the phase  $\beta$  [18].

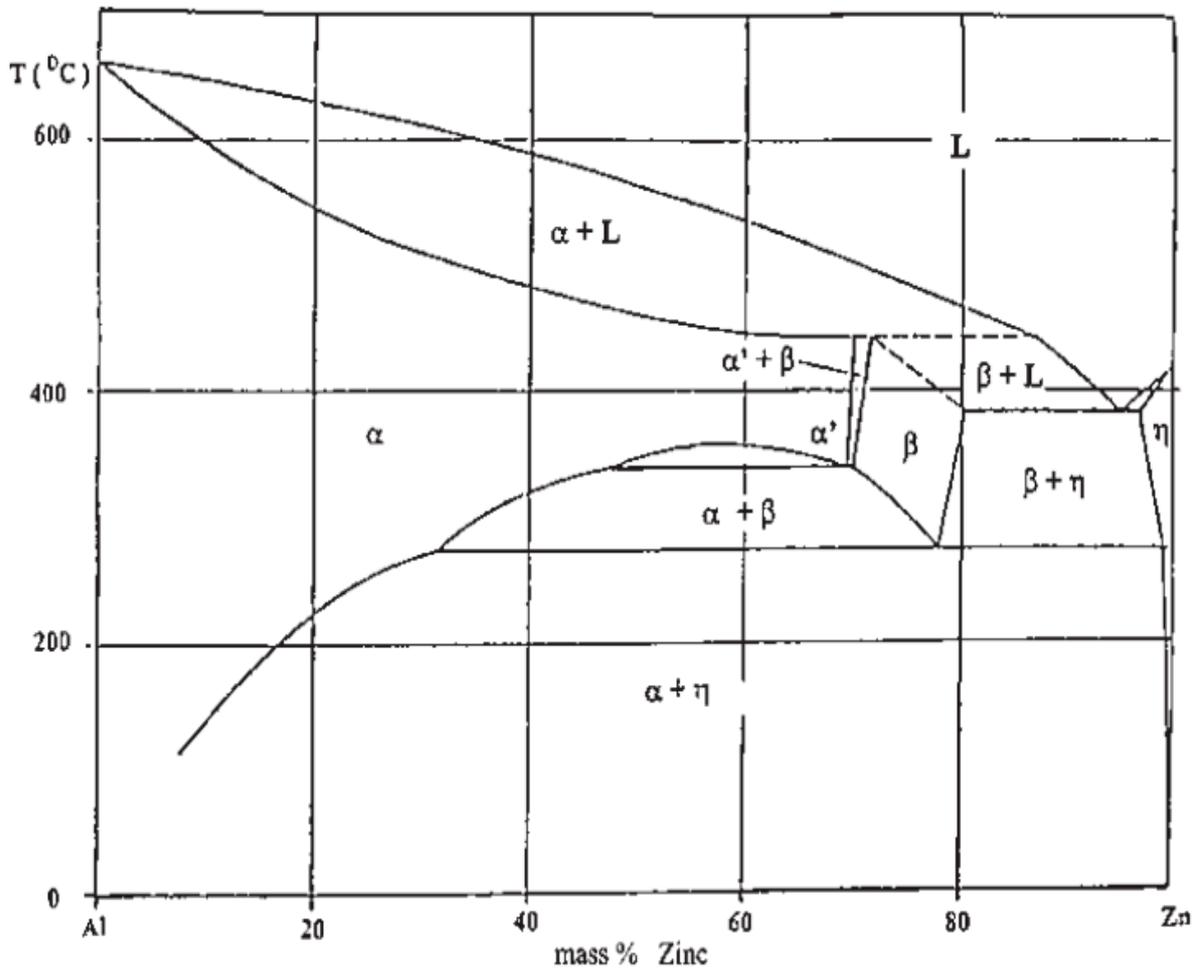


Figure 15: Phase diagram of binary Zn-Al alloy [27]

## 2.6 Post-Braze Heat Treatment- Solution Heat Treatment

As shown before, 7xxx series of wrought aluminium alloys are heat treatable for strengthening their mechanism. Thus, heat treatment is performed for both half of the Al-7046 sheets and AFS composites to analyze effects of the heat treatment while comparing with the Al-7046 sheets and AFS composites which heat treatment is not performed.

The wrought heat treatable 7xxx alloys are responsive to precipitation hardening and can obtain higher strength levels, approaching tensile strengths of 690 MPa (100 ksi). The 7xxx alloys can be naturally aged but are not because they are not stable if aged at room temperature; that is, their strength will gradually increase with increasing time and can continue to do so for years. Therefore, all 7xxx alloys are artificially aged to produce a stable alloy [9].

Precipitation hardening is used extensively to strengthen aluminium alloys. In precipitation hardening, an alloy is heated to a high enough temperature to take a significant amount of an alloying element into solid solution. It is then rapidly cooled (quenched) to room temperature, trapping the alloying elements in solution. On reheating to an intermediate temperature, the host metal rejects the alloying element in the form of fine precipitates that create matrix strains in the lattice. These fine precipitate particles act as barriers to the motion of dislocations and provide resistance to slip, thereby increasing the strength and hardness [9].

Precipitation hardening consists of three steps:

### 1. Solution heat treating

The first step is to produce a supersaturated solid solution. Solution heat treating is accomplished by heating the alloy to a high enough temperature and soaking for a sufficient time to achieve a nearly homogeneous solid solution. Nominal commercial solution heat treating temperatures are determined by the composition limits of the alloy, with an allowance for unintentional temperature variations. The time at temperature required for solution treating should be long enough for diffusion to occur to produce a supersaturated solid solution. The time required can be less than a minute for thin sheet to as much as 20 h for large inhomogeneous castings [9].

### 2. Rapid quenching

During quenching, it is important to cool the alloy quickly enough so that the supersaturated solid solution is maintained on cooling to room temperature. On the other hand, rapid quenching often results in high residual stresses and part distortion, so less drastic quenches frequently are used with some sacrifice in properties to minimize distortion [9].

To avoid appreciable precipitation during the quenching process, two requirements must be met [9];

- The time required to transfer the part from the furnace to the quench medium must be short enough to preclude slow cooling in the temperature range where very rapid precipitation can occur
- The cooling-rate capacity of the quench medium must be such that little or no precipitation occurs during cooling. For example, if a quench tank is used, the tank must be large enough that there is not a significant rise in the tank temperature during quenching [9]

Aluminium alloy parts are usually tank quenched in cold water to provide the optimum strength and corrosion resistance. For plate stock that is heat treated at the mill, quenching is conducted with high-velocity sprays of cold water. For parts with complex shapes that are subject to distortion, milder quenches are often used, such as aqueous solutions of poly-alkaline glycol or hot water maintained at 65°C to 80°C. Immediately after quenching, most aluminium alloys are nearly as ductile as they are in the annealed condition. Thus, straightening and forming operations are often conducted in the as-quenched temper. Because precipitation hardening will occur at room temperature, forming or straightening is usually conducted as soon as possible after quenching. Forming and straightening operations vary in degree from minor corrections of warp to complete forming of complex parts from solution-treated flat blanks. In either case, the plastic deformation must be completed before an appreciable amount of precipitation hardening takes place [9].

### 3. Aging

After solution treatment and quenching, hardening is achieved by aging at room temperature (natural aging) or by reheating to an intermediate temperature (artificial aging). In some alloys, sufficient precipitation occurs in a few days at room temperature to yield stable products with properties that are adequate for many applications. These alloys are sometimes artificially aged to provide increased strength and hardness in wrought or cast products. Other alloys with slow precipitation reactions at room temperature are always artificially aged [9].

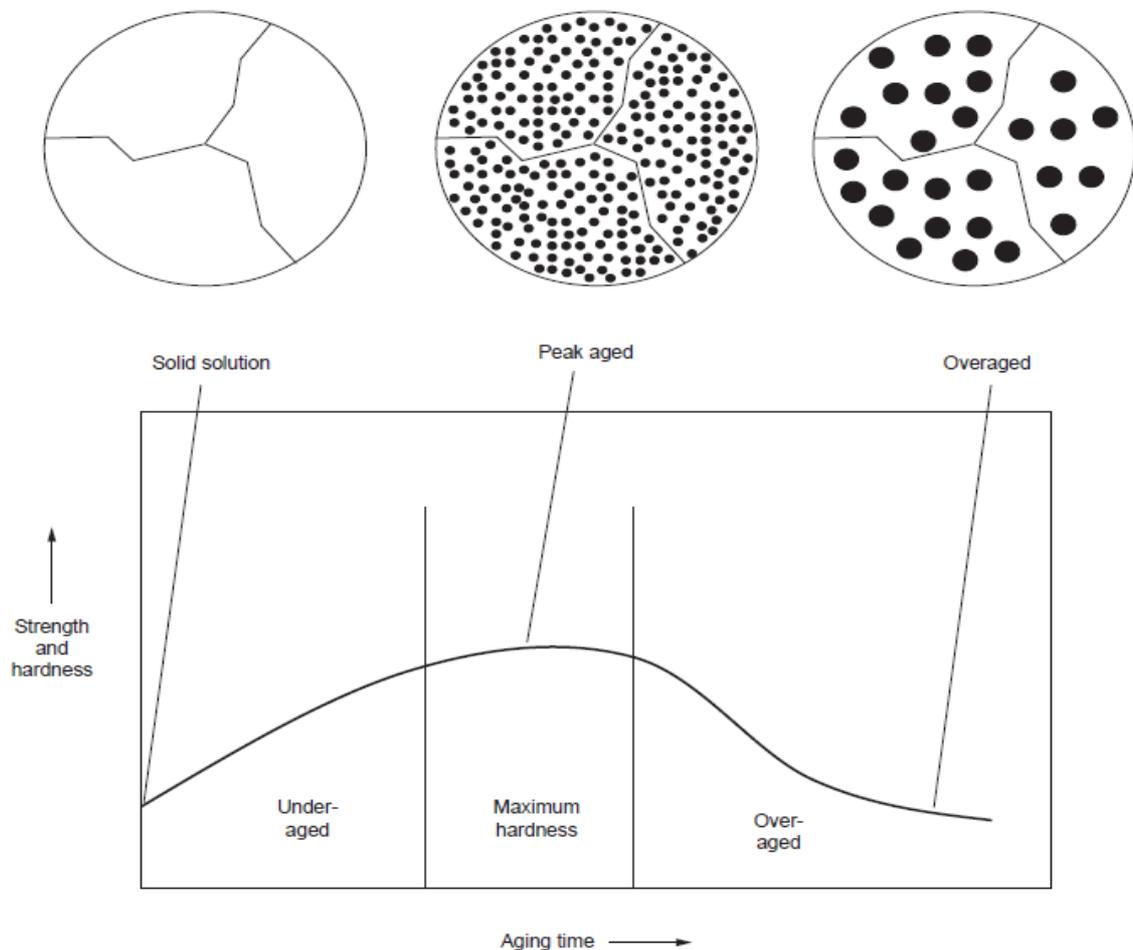


Figure 16: Typical aging curve for aluminium alloys [9]

- a. **Artificial Aging:** The wrought heat treatable 7xxx alloys are more responsive to precipitation hardening and can obtain even higher strength levels. These alloys are based on the Al-Zn-Mg(-Cu) system. The 7xxx alloys can be naturally aged but are not, because they are unstable when aged at room temperature; that is, their strength will gradually increase with increasing time and can continue to do so for years. Therefore, all 7xxx alloys are artificially aged to produce stable alloys. For aluminium alloys, solution treating, quenching, and artificial aging is designated as the T6 temper. This temper provides the highest strength levels that can be obtained, as illustrated for 7075-T6 aluminium in Figure 17 [9].

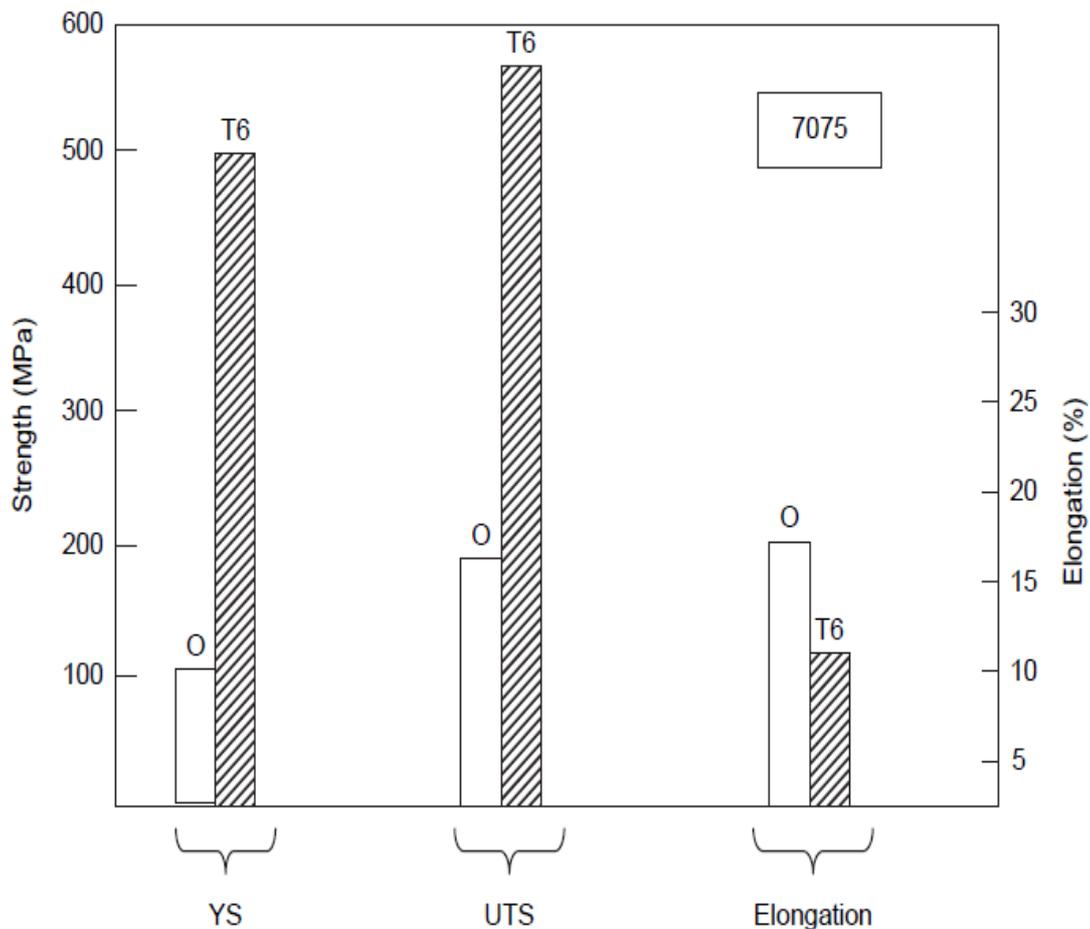


Figure 17: Effects of T6 heat treatment on 7075 Al. YS, yield strength; UTS, ultimate tensile strength [9]

The best combination of strength and corrosion resistance is produced by the T77 temper, which is a form of what is known as a retrogression and over-aging treatment. A summary of the effects of different tempers on 7xxx alloys is shown in Figure 18 [9].

### Solution Heat Treated & Aged Tempers

- **T6xx (~0.080 in. max.)**
  - Highest tensile strength
  - Possible exfoliation corrosion
- **T76xx (~0.250 in. max.)**
  - Medium tensile strength
  - Best exfoliation resistance
- **T73xx/T74xx (~0.250+ in.)**
  - Lower tensile strength
  - Best stress corrosion resistance
- **Stress relieved tempers**
  - Eliminates residual stresses
  - Txx51 - Stress relieved by stretching
  - Txx52 - Stress relieved by compression

- T6 Peak Aged
- T77 Retrogression and Overaging
- T79 Very Limited Overaging
- T76 Overaged to Achieve Enhanced Exfoliation Resistance
- T74 Overaged to Achieve Enhanced Exfoliation and SCC Resistance
- T73 Overaged to Achieve Optimum Exfoliation and SCC Resistance

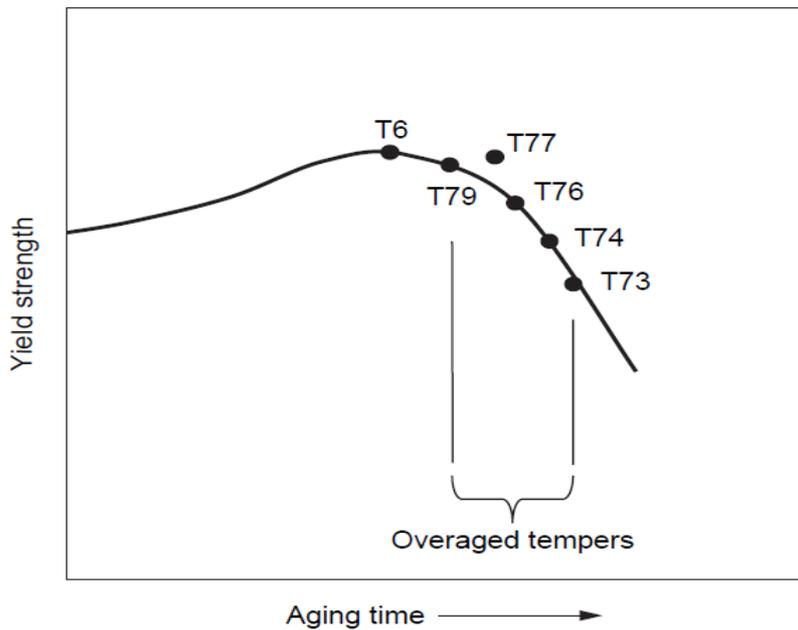


Figure 18: Effects of different tempers on 7xxx alloys [9]

According to improvement on Al-7075 shown before, it was decided to perform solution heat treatment to Al-7046 sheets in order to re-gain and increase strength, hardness after brazing joining process which during this process the cooling rate is low and it gives negative features for Al sheets and AFS composites. And rapid water quenching was decided to perform directly after solution heat treatment.

# CHAPTER 3. EXPERIMENTAL WORK

Experimental work done for the thesis is explained in this chapter with all the processes implemented for producing AFS composites and during all the processes details were given about materials used in this work, joining method and activity, heat treatments which is performed to AFS composite and Al sheets.

After the production of AFS composites, characterization methods were performed to the AFS composites and Al sheets such as optical microscopy, scanning electron microscopy (SEM), energy dispersive spectrometry (EDS), 3 point bending test and micro hardness test. All the information about how to perform these tests was explained.

## 3.1 1st Batch of Al-7046 Alloys

Initially, composition of Al-7046 alloy was investigated and seen that the main alloying element is Zn around 6.6-7.6%. Also braze joining heat treatment method and preparation processes for the Al sheets and Al-foam were decided for producing AFS composites. The next decision was taken for selection of brazing joining material, firstly pure Zinc was selected to use due to mainly its suitability to Al-7046 and secondly Zn2%Al was selected.

As it is seen on literature that yield strength, ultimate tensile strength, hardness and mechanical properties of the Aluminium sheets decrease after having braze joining heat treatment, mostly the reason is long cooling time. Also our Al-7046 is in the heat-treatable wrought alloys group, thus there is chance to gain all the mechanical properties with another heat treatment which is the following one to the braze joining heat treatment.

It is possible to gain these important mechanical properties with the proper heat treatment so called solution heat treatment. As literature recommending this heat treatment, I firstly performed this heat treatment to the aluminium plates for witnessing the increase of the strength and hardness. For comparing the mechanical properties with and without solution heat treatment, I obtained a plate of Al-7046 as received without doing any processes, a plate of Al-7046 which had brazing heat treatment for the simulation of producing AFS composites and the lastly a plate of Al-7046 which had solution heat treatment after brazing heat treatment. After characterization methods were performed such as optical microscopy, SEM, EDS, 3 point bending and micro hardness to the 3 different group of Al-7046 alloy.

It is explained all the preparation methods and processes for the 1<sup>st</sup> batch of Al-7046 alloy on the following title for understanding the importance of solution heat treatment in terms of strength, hardness, microstructure.

The first experiment is so called 1<sup>st</sup> batch of Al-7046 alloys. I performed this first experiment in order to see how much I can gain the strength and hardness after decrease due to brazing heat treatment. So, the processes for obtaining all the desire experimental information are the following.

Briefly, the steps are as following;

1. Brazing heat treatment for the 2 plates of Al-7046 alloys
2. Solution heat treatment for one plate of Al-7046 which already had brazing heat treatment
3. Leave one plate of Al-7046 as received for comparison
4. Then obtaining, one Al-7046 plate as received, one Al-7046 plate with brazing heat treated and one Al-7046 plate with solution heat treated
5. Prepare these 3 different plates of Al-7046 for the characterization methods
6. Cutting from the 3 different Al-7046 plates the samples with the dimension of 1 mm to 1mm for the tests of scanning electron microscope, energy dispersive spectrometry, micro hardness and optical microscope
7. Cutting from the 3 different plates again total 9 samples with the dimension of 60 mm to 20 mm for 3 point bending test
8. Performing 3 point bending test for 9 samples, 3 samples from each group
9. Gloss coating by epoxy resin and hardener for the 1 mm to 1 mm samples from 3 different group of Al-7046 for SEM, EDS, micro hardness and optical microscope
10. Following to epoxy resin and hardener processes, polishing process to coated samples to have bright surface and eliminate the scratches, oxidation layers
11. Performing optical microscope after polishing
12. Applying silver liquid on the surface of epoxy resin coated samples for getting the electric connection from the down-side to up-side for SEM, EDS tests.
13. As final, performing Vickers hardness test to these 3 samples

### **3.1.1 Brazing Heat Treatment**

The minimum temperature is 450 °C for performing brazing bonding heat treatment thus it was decided to implement this temperature firstly. Also, another important points for deciding the temperature were it should be less then melting point of the Al-7046 alloys which is around 607÷649 °C and it should be efficiently more than the melting point of the brazing joining material which is pure Zn and Zn2%Al in order to have perfect joining. The melting point is 419.5 °C for pure Zn and melting point for Zn2%Al is 376÷385 °C. Additionally, the temperature should be below the eutectoid temperature of the alloys in case of not having second phase precipitations. Thus, the initial temperature is decided to perform is 450 °C for the Al-7046 plates.

Next step was decision of the heating rate, cooling rate and dwell time for the brazing heat treatment. In order to have perfectly joint in this process, cooling rate should not be fast and it is also recommended to 10 °C per minute. This rate is efficiently enough for obtaining good joints. On the other hand, obtaining good stabile melt of the brazing joining element and permeation with the Al sides need some time so there is needed to have short dwell time on the maximum temperature before starting to cool down. This dwell time should not be long due to degradation of the Al and Zn because the temperature is on the maximum level of the process. It is decided firstly 10 minutes for the dwell time.

As more experiments performed for the production of AFS composite, this time would be changed depending on the results. Also, the heating rate should not be so slow or so fast for degradation, crack up and distortion. It was also recommended to perform with 12 °C per minute for production of AFS composites and brazing heat treatment process. These two rates, heating rate and cooling rate, they are constant and I am not able to change them. The only 2 parameters I can modify are dwell time and brazing heat treatment temperature.

Later on, during the experiments for finding the brazing temperature of AFS composites, I changed the brazing temperature to different values to figure out the best suitable brazing temperature. In the beginning of my thesis, it was provided 3 plates of as received Al-7046 to me. As explained before, I need 3 different groups of this Al-7046 plates;

- 1. As received Al-7046, AR
- 2. Brazed heat treated Al-7046, BH
- 3. Solution heat treated Al-7046, SH

Solution heat treatment is the continuously following heat treatment after brazing heat treatment thus I needed to conduct brazing heat treatment for 2 pieces of as received Al-7046 plates. After deciding all the parameters for starting up the brazing heat treatment, it was time to conduct the process with the following diagram in figure 19.

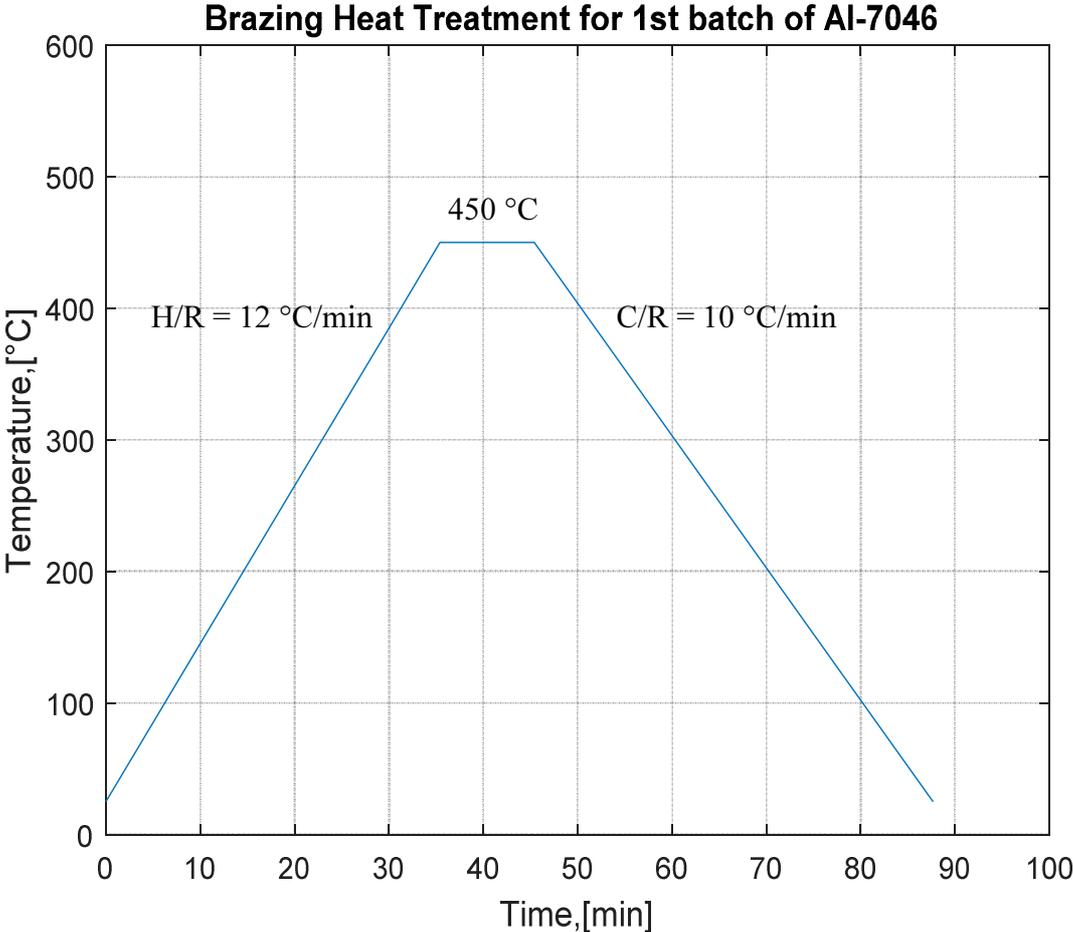


Figure 19: Brazing heat treatment for Al-7046 plates

Brazing heat treatment was performed with the parameters on above to the 2 Al-7046 plates in the furnace which name is Carbolite CWF 1300, figure 20. And two Al as received plates were located in the furnace in the figure 21.



Figure 20: Brazing heat treatment furnace for 1<sup>st</sup> batch of Al-7046 plates



Figure 21: Locating the Al as received plates in the furnace

As following to start up, the furnace was switched on from the green button and all the data was set on the screen as brazing temperature 450 °C, heating rate 12 °C per minute, cooling rate 10 °C per minute and dwell time 10 minutes.

As soon as it was done with the setup all the required data, it was pressed to run on the set-up screen, figure 22.

Then as following it was switched on the heating from the red button and the furnace was started to heat up immediately. The total process took around 2 hours to finish.



Figure 22: Setting up for the furnace

When the brazing heat treatment to Al-7046 plates was over, firstly the heating was turned down from the red button and subsequently the power was also turned down from the green button. Furnace gloves were wear for the protection against the heat in case of the furnace was still hot then the door of the furnace was opened and plates were carried out of the furnace by metal pincer. Finally, the brazing heat treatment for Al-7046 plates was over with the last step.

### 3.1.2 Solution Heat Treatment

7xxx series of wrought aluminium alloys are heat treatable for strengthening their mechanism and responsive to precipitation hardening and can obtain higher strength levels such as tensile strengths of 690 MPa.

Solution heat treatment is one of the heat treatments methods for Al-7046 alloys to increase their yield strength, ultimate tensile strength, hardness and mechanical properties. For that case, the Al-7046 plate, which was already had brazing heat treatment, was heated to high enough temperature to take a significant amount of an alloying element into the solid solution.

The fine precipitates were created matrix strains in the lattice and these particles acting against the motion of dislocations which provide strength and hardness.

Heating rate of the solution heat treatment should not be so slow or so fast for degradation, crack up and distortion, the same for brazing heat treatment. It was always recommended to perform with 12 °C per minute for heating up.

Also, the temperature for the solution heat treatment should be less than the brazing heat treatment due to the solution heat treatment is kind of recovery heat treatment for the mechanical parameters we lost during the brazing heat treatment.

On the other hand, it is not proper to destroy the micro structures and joint we had after brazing heat treatment while going up to higher temperature than brazing temperature, thus it was decided to perform 370°C for solution heat treatment which less than the brazing heat treatment temperature 450°C, also, less than the melting temperatures of pure Zinc and Zn2%Al joining alloys.

Thus, the temperature 370 °C guarantees the joint structure which is obtain by brazing heat treatment without having any melt of the parts.

The dwell time of the solution heat treatment is also quite important that it should be long enough for diffusion to occur to produce a supersaturated solid solution. On the literature, it is said that it can be less than a minute to 20 hours depending on the plate thickness and its size.

Considering the 10 cm to 15 cm dimensions of Al-7046 plate, firstly it was decided to conduct dwell time for 10 hours in case of obtaining good level of supersaturated solid solution.

We are also free to modify the temperature and the dwell time for the solution heat treatment. Later on, after having some experiments, the dwell time was reduced from 10 hours to 3 hours. Because, there is no any certainly designated dwell time for the process. It is obtain by experiments and considering the results depending on your sample.

Solution heat treatment is the subsequent process after the brazing heat treatment. As following to remove 2 pieces Al-7046 plates from Carbolite CWF 1300 furnace, one piece of the brazed heat treated Al-7046 plate is conducted to solution heat treatment with the parameters above.

As final of this treatment, I obtained total 3 Al-7046 plates that one of them was as received, another one was brazed heat treated and the last one was solution heat treated.

After deciding all the parameters for starting up the solution heat treatment, it was time to conduct the process with the following diagram in figure 23. The first curves part of the figure 23 represents the brazing heat treatment and subsequently the second curves part represents the solution heat treatment and water quench.

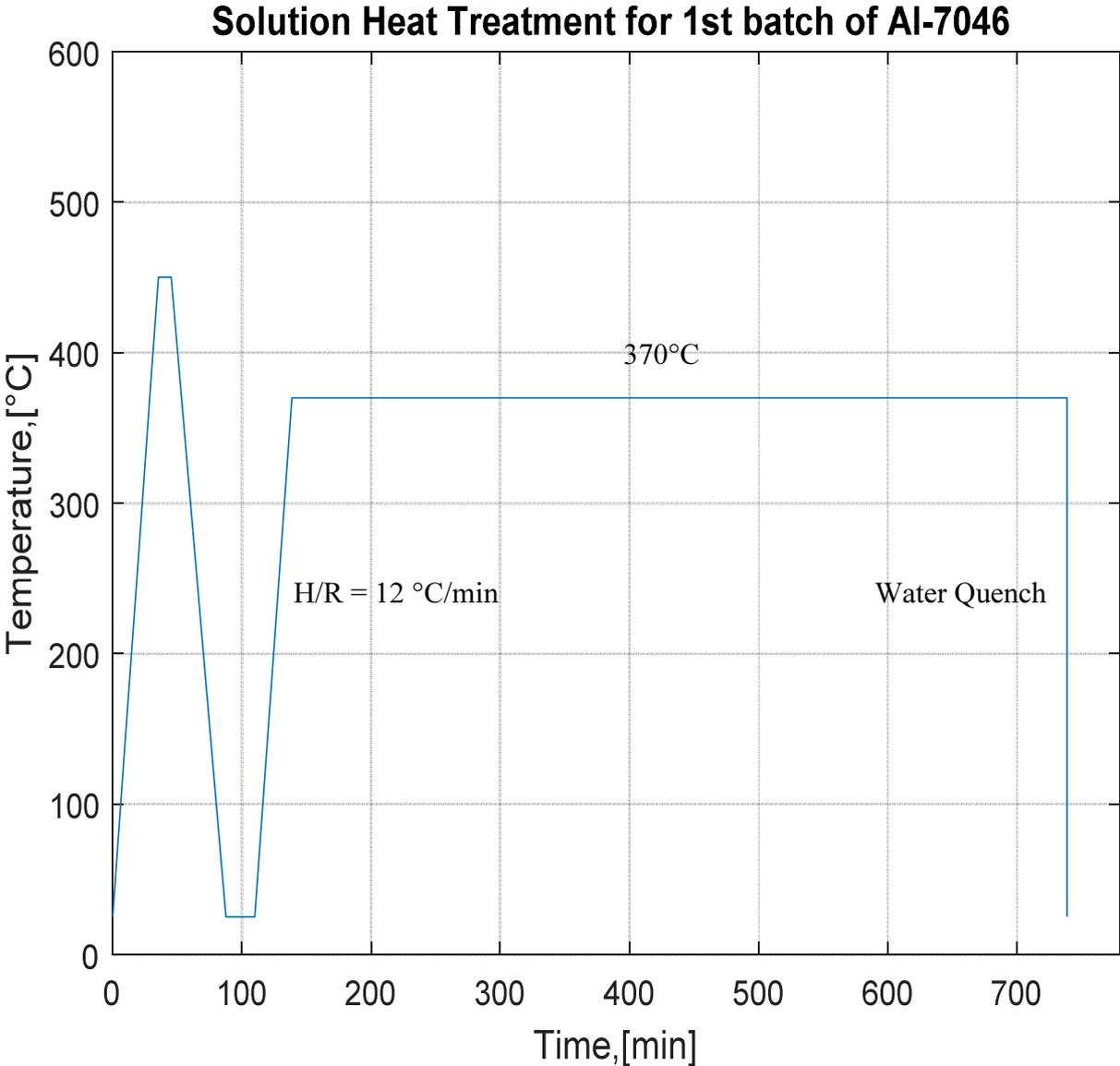


Figure 23: Solution heat treatment for Al-7046 plates

Solution heat treatment was performed with the parameters on above to the one piece of Al-7046 plates in the furnace which name is OVMAT 2009, figure 24. And one piece Al brazed heat treated plates was located in the furnace.



Figure 24: Solution heat treatment furnace for 1<sup>st</sup> batch of Al-7046 plates

After achieving good level of supersaturated solid solution in the end of the dwell time for 10 hours, instead of cooling down in the furnace, rapid water quenching was performed in order to have all these supersaturated solid solution in the room temperature.

As following the solution heat treatment, rapid water quenching is needed for maintaining the supersaturated solid solution in the room temperature. It is really important that you might have high residual stresses or part distortion after quenching if it is not performed correctly.

For avoiding these side effects, the time to transfer part from the furnace to the water quenching medium must be as short as possible and quenching must be uniformly done for all the points on the part at the same time homogeneously.

As shown in the figure 25, water quenching was performed directly when the dwell time was over. The plate was transferred in the quenching medium by a metal pincer.



Figure 25: Water quenching

Because of the long dwell time, 10 hours, all these processes took around 11 hours including setting up, water quenching. Special care was done while transferring the plate inside the quenching medium with furnace gloves and laboratory uniform.

Following the completion of solution heat treatment, 3 different Al-7046 plates were obtained and they were ready to be cut in small dimensions for characterization methods such as 3 point bending, micro hardness, SEM, EDS and optical microscopy.

Also, every time before doing any furnace processes, it is highly essential to know the calibration of the furnace. Before I was conducting the all heat treatments, furnaces were calibrated recently. At the same time, it is also essential to obtain clean environment inside the furnace without any remaining particles from the one previous experiment.

Now, all the Al plates were ready to be cut for further steps.

### 3.1.3 Gloss Coating

Firstly, for gloss coating, the liquid mixture was obtained by mixing epoxy resin and epoxy hardener with the rates of 12 units resin to 3 units hardener. There is 1 to 4 unit rate each other. That liquid mixture should be well mixed before the gloss coating.

The samples were put on the bottom cover of the plastic housing containers and taped for holding still while pouring the liquid mixture inside the plastic housing containers. There is a key trick that when the liquid mixture is hard after 8÷10 hours, it is really difficult to remove the gloss coated sample from the plastic containers.

To avoid this difficulty, just a little liquid soap was applied on the surfaces of the plastic containers where they have contact with the liquid mixture. At that case, it is easier to remove the gloss coated samples out from the plastic containers.

As shown in the figure 26, after pouring the liquid mixture by syringe inside the plastic container, it was rested around 8÷10 hours for hardening and being one solid part.

As following the hardening and having solid parts, they were removed gently from the plastic housing containers with good care and samples were ready for the next operation so called polishing for the aim of having bright surfaces without scratches and without any oxide layers, Aluminium and its alloys are sensitive to oxidation easily in the air, in order to analyze the micro structure by optical microscope, EDS, SEM.

Now, the samples are ready for the polishing operation.

Shown in the figure 26, there are 2 samples of as received Al-7046 which was represented with number 1, 2 samples of brazed heat treated Al-7046 which was represented with number 2 and 2 samples of solution heat treated which was represented with number 3.



Figure 26: Gloss coated samples in the mixture of epoxy resin and hardener

### 3.1.4 Polishing Operation

Polishing is the final finishing process for having smooth shiny surfaces also eliminating the oxide layers on the samples. It is done by abrasive plates with different surface roughness numbers. Aluminium and its alloys are quite sensitive and soft thus it is really difficult to polish them perfectly, also it takes long time and effort.

Firstly, I started to polish my 6 samples with the 800 number of abrasive plate then subsequently 1200 number of abrasive plate, 2000 and 4000. When the numbers of the abrasive plates increase, it means that the surface roughness decreases. After removing all the scratches on the surface, as final for aluminium and its alloys, it is also practical to use diamond liquid during polishing. This diamond liquid gives really good brightness on the surface.

Polishing is done with water supply due to polishing is an operation that some particles emerge to the air and it is hazardous to inhale them so water helps to keep these emerged particles inside and, also, it helps for cooling the sample during polishing. Another important point during polishing is keeping the sample fixed position on the abrasive plate in order to have flat surfaces. It is not practical to use the force on the sample against to plate, it might cause not having flat surfaces and it is big problem during micro hardness test, optical microscopy and so on. Instead of pushing the sample on the abrasive blade, just holding it on a fixed position is always better to have flat surfaces, also for goodness of the following further steps.

Polishing was done with the machine, as shown in the figure 27. It is shown that water supply is on, abrasive blade rotates and gloss epoxy coated sample on my hand while polishing operation.

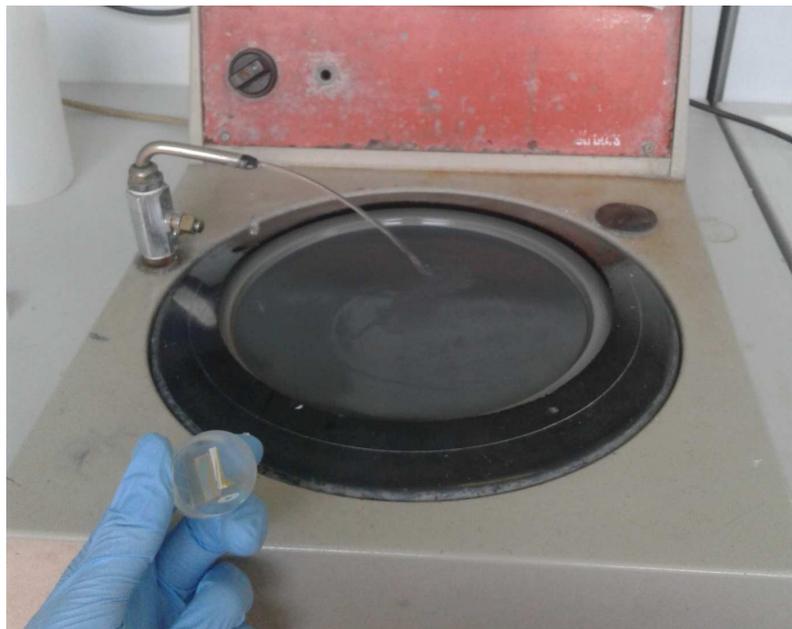


Figure 27: Polishing machine, polishing 1<sup>st</sup> batch of Al-7046 samples

Total 6 samples, 2 of them as received number 1, 2 of them brazed heat treated number 2 and 2 of them solution heat treated number 3, were polished by different abrasive plates starting with the number 800 and subsequently 1200, 2000 and 4000 number of abrasive plates. Following the 4000 number, all the samples were polished with diamond liquid to give final brightness.

When polished was done, it should be given good care of the samples not to touch the polished surfaces. It is very easy to lose all the brightness and smooth surface with little touch of something on the surface of Al surface because it is soft material.

Also, it can be oxidized very easily on time in the normal environment, thus it is essential to perform following operations without spending any time. So, analyzes of the micro structures by optical microscopy were performed as quick as possible after all the 6 samples were done with polishing.

### **3.1.5 Optical Microscopy**

When there are no more scratches on the surfaces of the samples and they are bright enough, the images of the microstructures were taken by the optical microscope with different magnitudes for each sample.

Firstly, it was started with the magnitude of 100-1.25 for as received Al-7046 gloss coated sample, number 1, and 5 microstructure images were saved, then subsequently 5 micro structure images were saved for each different magnitudes for 100-1.88 and 50-1.25. In total, I had 15 images for these 3 different magnitudes for as received, number 1.

The same processes were conducted both for brazed heat treated Al-7046 gloss coated sample, number 2, and solution heat treated sample, number 3, with the 3 different magnitudes.

It was obtained totally 45 images for these 3 different kinds of Al-7046 samples, number 1, 2 and 3, with 3 different magnitudes, 100-1.25, 100-1.88 and 50-1.25. Microstructures were investigated and analysed if they had second phase or precipitation hardening in the structure.

Mainly optical microscope consists on lenses, eyepieces, light source and camera.

Briefly working principle, light from an incandescent source is aimed toward a lens beneath the stage called the condenser, through the specimen, through an objective lens, and to the eye through a second magnifying lens, the ocular or eyepiece. The condenser is used to focus light on the specimen through an opening in the stage. After passing through the specimen, the light is displayed to the eye with an apparent field that is much larger than the area illuminated [19].

However, there are some disadvantages of OM such as low resolution and low magnitudes. That's the reason, I performed scanning electron microscopy (SEM) subsequently to optical microscopy.

### 3.1.6 Scanning Electron Microscopy (SEM)

Scanning electron microscopy is the following operation after optical microscopy there are some preparation procedures for the samples to make it ready for SEM. Firstly SEM is conducted by electric field so it is obligation to get electric connection between the samples and the microscope. There are also metal clamping tools which help to hold still the samples inside the microscope. For getting the electric circuit close from bottom side to top side of the gloss coated samples, silver liquid was applied on the surface as shown in the picture 28. The white colour on the edge of the metal is dried silver liquid. Number 1 represents as received Al-7046, number 2 represents brazed heat treated and number 3 solution heat treated.



Figure 28: Silver liquid was applied on the gloss coated samples

Secondly, before conducting SEM, it is always better to have samples dry that not seeing little water drops on the surface with SEM. To avoid this situation, samples were put inside the furnace with low temperature around 10 minutes to make them really dry. This low temperature furnace procedure was applied in order to get good results from SEM without seeing little water particulars on the analysis. SEM and EDS analyzes were performed on the microscope which the name is Zeiss Merlin as shown in the figure 29. Samples were put inside the specimen chamber and vacuum was turned on in the chamber.



Figure 29: SEM, Zeiss Merlin

Accordingly to the desire magnitudes and desire points on the surface which it was needed to analyze, arrangements were done by adjustment control panels and different magnitudes, to get the microstructure images with better resolution and better magnitude than the optical microscopy. Scanning electron microscope works the same principles of optical microscope however uses electrons instead of light, electrical field.

Briefly, in scanning electron microscopy (SEM) an electron beam is focused into a small probe and is raster across the surface of a specimen. Several interactions with the sample that result in the emission of electrons or photons occur as the electrons penetrate the surface. These emitted particles can be collected with the appropriate detector to yield valuable information about the material. The most immediate result of observation in the scanning electron microscope is that it displays the shape of the sample. The resolution is determined by beam diameter [20]. It is shown on the figure 30 that the parts of the SEM and their configuration. The detailed working diagram is represented in figure 31.

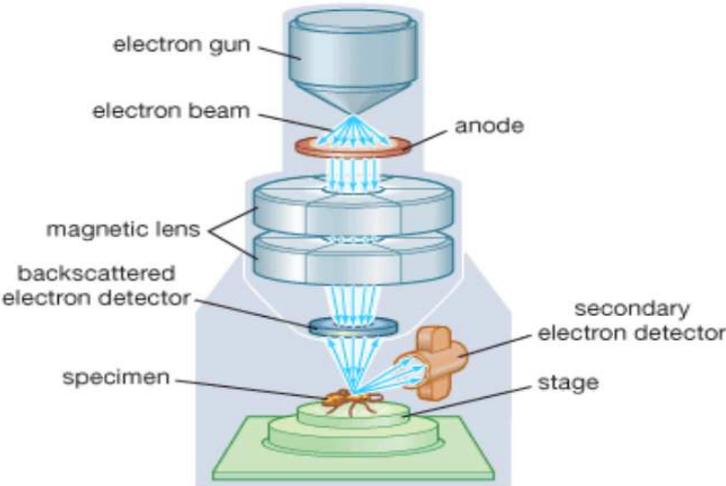


Figure 30: Basic configuration of SEM [20]

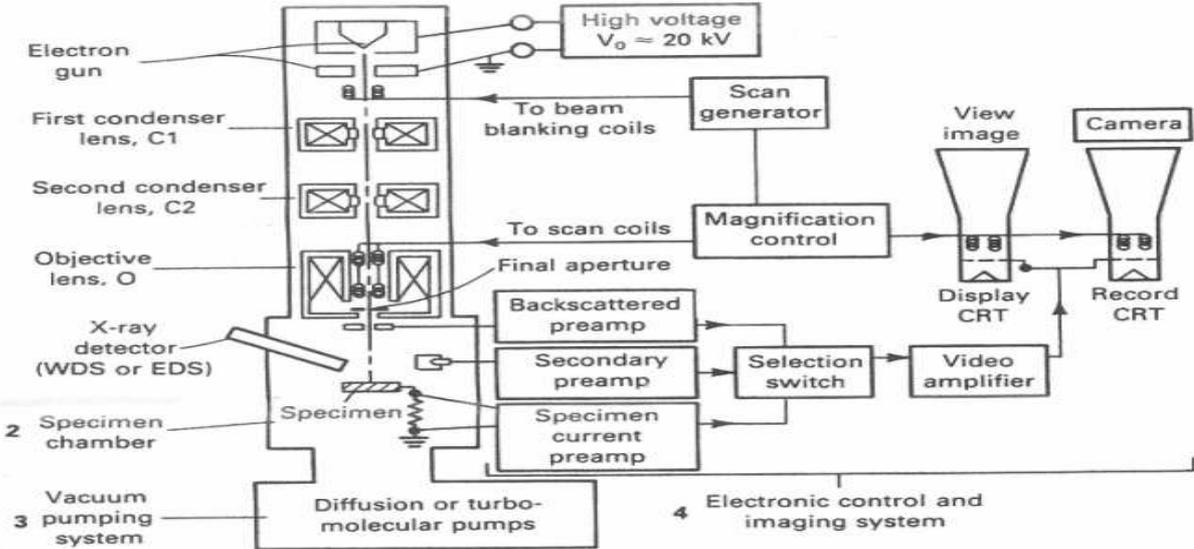


Figure 31: Detailed working diagram of SEM [20]

### 3.1.7 Energy Dispersive Spectrometry (EDS)

The Zeiss Merlin machine has got the functions of making EDS and SEM analyzes together due to X-ray spectrometer is added on it. After achieving electron images with SEM, it is also possible to have element mapping, even point analysis and area analysis at the same time. These 2 functions were together in one instrument.

When the desired electron image is obtained for a point or an area, it is possible to have the chemical analysis for seeing all the particular elements and their relative proportions on selected position. As shown on the figure 32, desired spectrum position was selected on the electron image then it was shown the particular elements on chemical analysis.

With EDS analysis, I obtained the chemical structure of particular positions of these 3 different samples, as received ones, brazed heat treated and solution heat treated ones. When I had the results of the composition of the elements, I could make my investigation about the effects of the heat treatments in terms of chemical structures.

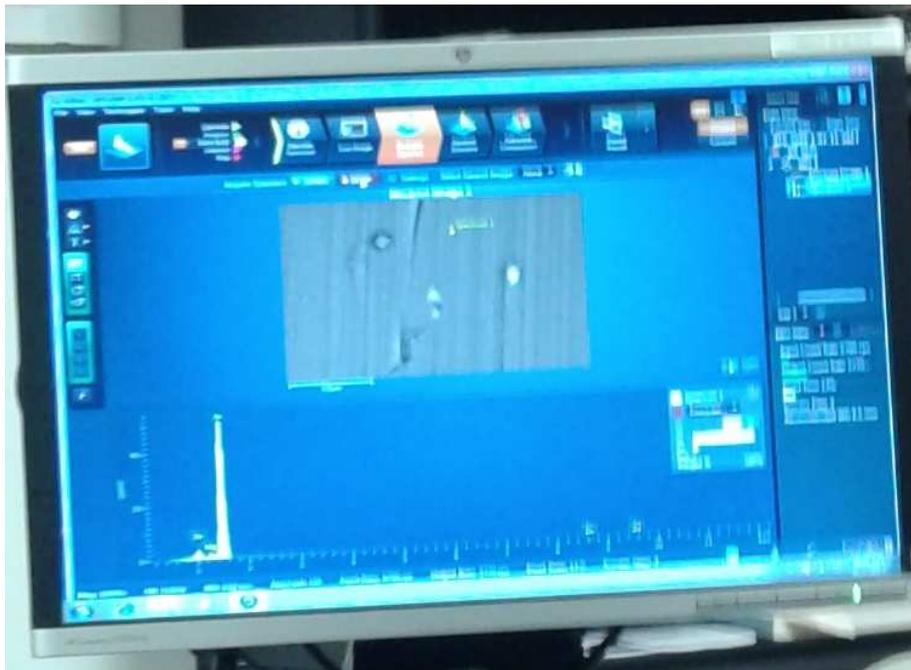


Figure 32: Computer screen for SEM and EDS analysis

EDS makes use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis. All elements from atomic number 4 (Be) to 92 (U) can be detected in principle, though not all instruments are equipped for 'light' elements ( $Z < 10$ ). Qualitative analysis involves the identification of the lines in the spectrum and is fairly straightforward owing to the simplicity of X-ray spectra [21].

Quantitative analysis (determination of the concentrations of the elements present) entails measuring line intensities for each element in the sample and for the same elements in calibration standards of known composition [21].

By scanning the beam in a television-like raster and displaying the intensity of a selected X-ray line, element distribution images or 'maps' can be produced. Also, images produced by electrons collected from the sample reveal surface topography or mean atomic number differences according to the mode selected [21].

X-ray spectral measurement is done with the method that the absorption of an individual X-ray photon by the detector leads to the ejection of a photoelectron which gives up its energy to the formation of electron hole pairs [22].

The electron-hole pairs in turn are swept away by an applied bias to form a charge pulse. The charge pulse is converted to a voltage pulse by a preamplifier. The signal is further amplified and “shaped” by a linear amplifier (pulse processor) and then passed on to a multi channel analyzer where the data is displayed as a histogram of intensity vs voltage [22].

The important point here is that the voltage pulse produced is proportional to the energy of the incoming X-ray photon [22]. The X-ray spectral measurement flow chart was shown on the figure 33. Relatively, when the energy of the X-ray photon is high, the voltage pulse peak is high as well.

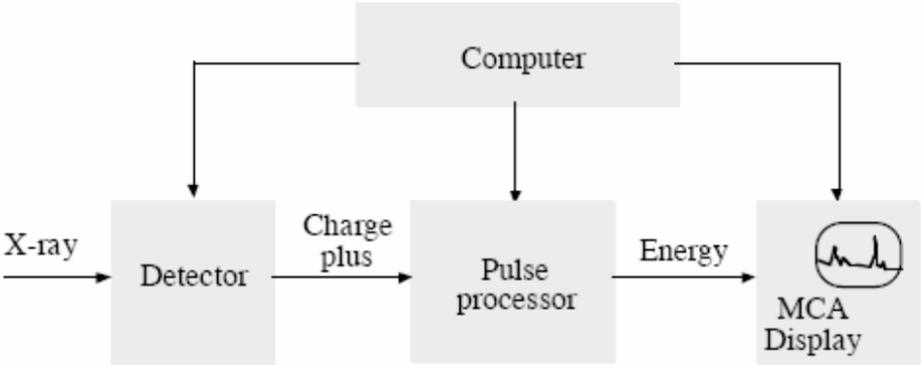


Figure 33: X-ray spectral measurement [22]

After obtaining all the electron images by SEM for investigating the microstructure, grain boundaries, second phases, precipitation of the phases, and then chemical analysis by EDS of these 3 different samples, as received number 1, brazed heat treated number 2, solution heat treated number 3, for comparing the dispersion of elements in the microstructure depending on the heat treatments effects as increase or decrease of the element percentages.

As following to SEM and EDS, the new process is micro hardness test in order to investigate the hardness of these 3 samples for finding the effect of heat treatments.

### 3.1.8 Micro Hardness Test

Vickers micro-hardness test was performed subsequently to SEM and EDS analyzes. The aim of this hardness test was to understand the effect of different heat treatments on the hardness of the samples. As usual, we have 3 different groups of samples such as, as received number 1, brazed heat treated number 2 and solution heat treated number 3.

Before starting the micro-hardness test, it is essential to have flat surfaces on the both sides of the sample. If there is an inclination on the surface, when the Vickers head comes to give the force on the sample, which is put on the instrument table, the Vickers head might slip on the surface and it is not able to create proper Vickers mark with proper depth because of the inclination. To avoid that slipping head problem, epoxy kitt was used in order to obtain flat surface on the bottom side of the sample for alignment, see the Figure 34.

Big attention should be taken while working with epoxy kitt due to its chemical structure, it is highly hazardous to inhale, gloves and mask should be worn and operation should be done inside the close laboratory cabinet under the ventilation system.

Epoxy kitt is like a paste and it should be mixed perfectly in short time and should apply to the bottom side of the gloss coated sample. Then, it should be pressed homogeneously in order to obtain flat surface on the bottom side and it becomes hard around 5 minutes. On Figure 35, it is shown that the epoxy was applied on the bottom surfaces of the 3 different groups of gloss coated samples in order to obtain flat bottom surfaces.



Figure 34: Epoxy kitt



Figure 35: Epoxy kitt was applied on the bottom surface of glossed coated samples

For Vickers micro-hardness test, I used the HX-1000 Remet hardness instrument which is directly connected to the computer and it is shown on the figure 36. The selected operation parameters were dwelling time 15 seconds and force 25gf.



Figure 36: Vickers micro-hardness instrument

The operation was conducted with these main steps;

1. Turning on hardness instrument and computer programme for hardness test
2. Transferring the sample on the hardness table and adjustment the point where I want to take the hardness value by 2 control handles which helps to move the table on desire location, both I could see the point with different magnitudes from computer programme or eyepiece on the instrument
3. Removing the plastic cover from Vickers head on the cylindrical rotating head and pushing the switch button
4. Now the Vickers head is the upward position on sample
5. Adjustment for the force, which applies during the test by left cylindrical control handle, and adjustment of the dwell time, which the Vickers head remains inside the sample during that time, from the control panel.
6. When all the data and location of the sample were ready, pressing start button for the operation
7. As soon as the operation over, automatically the cylindrical rotating head rotates and lens comes on the upward position so it is available to see the Vickers hardness mark on the sample by computer programme.
8. Specifying the 4 points on the edges of the Vickers hardness mark, programme automatically calculates the hardness value and it is shown on the computer screen
9. Then the process was repeated for all the samples

During the Vickers micro-hardness test, 7 hardness images with Vickers head marks were taken for each sample, thus the total number of images and data were 21 for 3 different groups of samples.

The Vickers hardness test method consists of indenting the test material with a diamond indenter, in the form of a pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a test force of between 1gf and 100kgf. The full load is normally applied for 10 to 15 seconds [23].

For my hardness tests, I applied 25 gf test force and 15 seconds dwell time with full load, as dwelling shown in the figure 37.

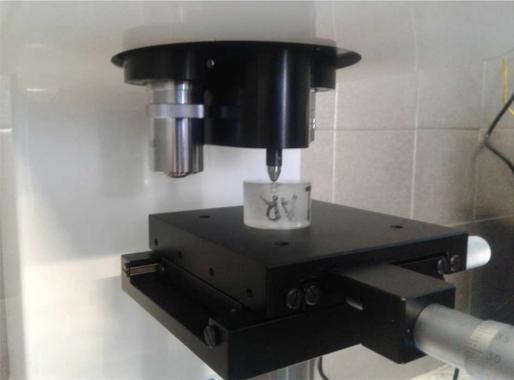


Figure 37: Dwelling for 15s with the max load 25gf to as received Al-7046 sample

The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surfaces of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of indentation [23], as shown on the following equation, also figure 38 represents the Vickers head and the remaining mark on the surface.

$$HV = \frac{2F \sin \frac{136^\circ}{2}}{d^2} \qquad HV = 1.854 \frac{F}{d^2} \text{ approximately} \qquad [23]$$

F = Load in kgf,  
d = Arithmetic mean of the two diagonals, d1 and d2 in mm  
HV = Vickers hardness

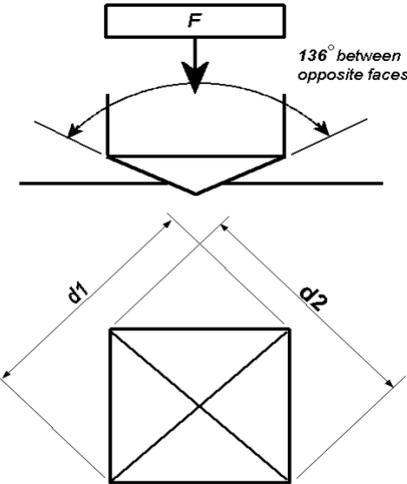


Figure 38: Vickers hardness mark with two diagonals [23]

### 3.1.9 3-Point Bending Test

As following to the cutting operation, I obtained total 9 samples with the dimension 60mm to 20mm from 3 groups that the group number one is as received Al-7046, group number two is brazed heat treated Al-7046 and the last group number 3 is solution heat treated Al-7046. As it is shown in the figure 39, its group number 2 and sample number 3 which represents brazed heat treated Al-7046 group and it is third sample from this group.



Figure 39: Brazed heat treated Al-7046 before 3 point bending

The three point bending test, figure 40, is a classical experiment in mechanics, used to measure the Young's modulus of a material in the shape of a beam. The beam, of length  $L$ , rests on two roller supports and is subject to a concentrated load  $P$  at its centre [24]. Span length 47 mm for bending tests.

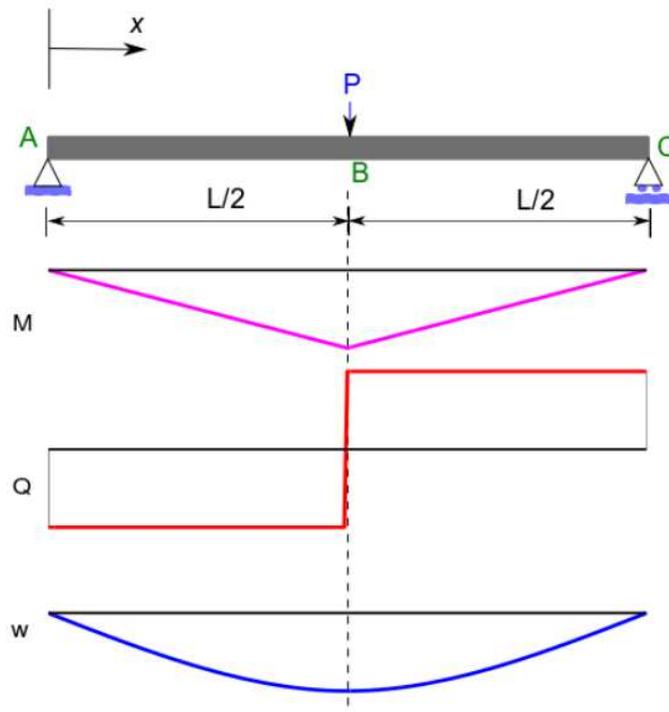


Figure 40: Schematic of the three point bend test (top), with graphs of bending moment  $M$ , shear  $Q$  and deflection  $w$  [24]

It can be shown (the Cambridge University Engineering Department Structures Data Book) that the deflection  $w_0$  at the centre of the beam is [24];

$$w_0 = \frac{PL^3}{48EI}$$

E is the Young's modulus

L is the span length

P is the concentrated load

I is the second moment of area defined by;

$$I = \frac{a^3b}{12}$$

a is the beam's depth

b is the beam's width

By measuring the central deflection  $w_0$  and the applied force P, and knowing the geometry of the beam and the experimental apparatus, it is possible to calculate the Young's modulus of the material [24].

Also the bending moment M was calculated with the following equation;

$$M = \frac{PL}{4}$$

L is the span length

P is the concentrated load

Maximum stress  $\sigma_{\max}$  for 3 point bending was calculated with the following equation;

$$\sigma_{\max} = \frac{3PL}{2ba^2}$$

L is the span length

P is the concentrated load

a is the beam's depth

b is the beam's width

Before starting to the 3 point bending test, some physical properties of the test samples were measured such as length, thickness, width and weight because width and thickness were the input of the bending test for the calculations, shown in previous page, which were done by computer software. It is also practical to get the data of the weights from these 3 groups in order to compare the effects of heat treatments on the weight.

For all the 3 point bending tests, deflection was set on maximum value 10 mm before starting to the test and I obtained some data such as modulus (MPa), maximum load (N) and maximum stress (MPa) end of the test by the computer software.

All the 3 point bending test was performed on the MTS Criterion Model 43 bending machine which has got 50 kN capacity with 2.108 mV/V sensitivity on the load cell. It is shown in the figure 41, and the loading speed was 4mm per minute for all the tests.



Figure 41: 3 point bending test machine

Subsequently all 9 samples were located on the support span, figure 42, and the maximum deflection value, width and depth values of the samples were inputted on the software programme then the test was conducted. When the deflection came to the maximum value, figure 43, which was 10 mm, the load was stopped and the test was over.

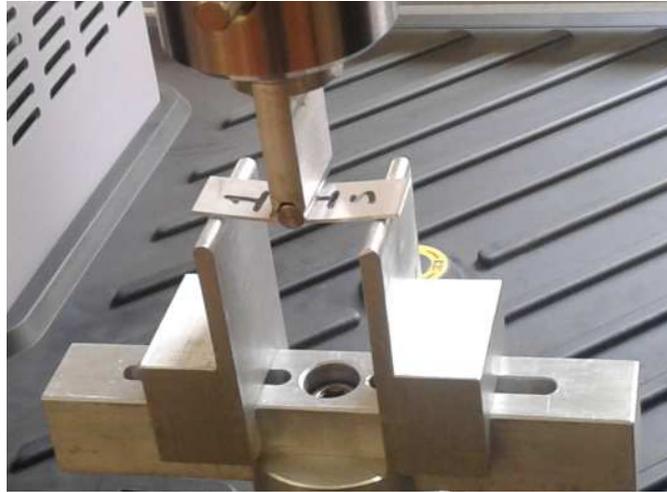


Figure 42: As received sample number 1 Al-7046 was located on the support span

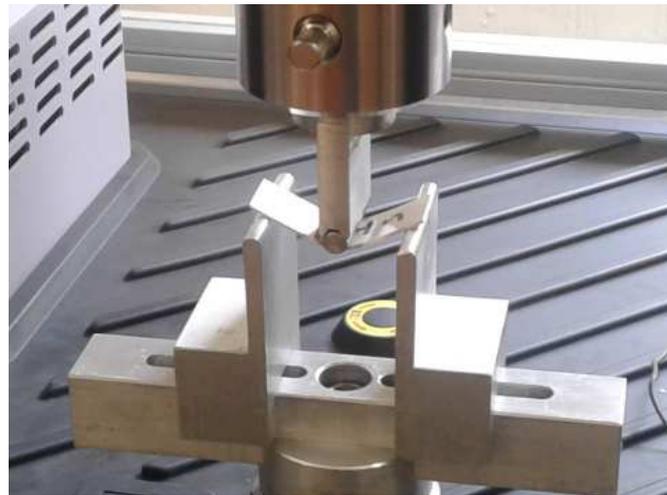


Figure 43: The sample when the deflection came to maximum value

End of the tests, computer programme gave the data about deflections and their corresponding loads, modulus and maximum stresses. After I obtained all the data, I drew load (N) vs cross head displacement (mm) chart for 3 pieces of as received Al-7046 samples group number 1, 3 pieces of brazed heat treated Al-7046 group number 2 and 3 pieces of solution heat treated Al-7046 samples group number 3.

All the load - cross head displacement curves were overlap each other in the same chart in order to compare the effect of the heat treatments to understand if I had increased mechanical properties while conducting additional heat treatment which was solution heat treatment, after brazing heat treatment.

## **3.2 Production of AFS Composite**

Production of AFS composites were done by braze joining method while joining 2 pieces of Al-7046 alloys with one piece of Al-foam, using pure Zn and Zn2%Al as brazing joint materials. As following to the joining operation, solution heat treatment was conducted for half of the produced AFS composites in order to investigate and compare the mechanical properties and microstructures of these composites if any improvement was succeeded.

In this section, preparation methods of the Al-7046 plates, Al-foams and brazing joint materials, pure Zn and Zn2%Al, furnaces and their equipments were explained for the production of AFS composites. Preparation methods were always the same for producing each AFS composite and they were repeated all the time.

After some trials of brazing operation with 2 pieces of Al-7046, 1 piece of Al-foam and pure Zn as joint material, the best joint was obtained for production of the AFS composite with the optimal furnace parameters. Then obtaining the optimal furnace parameters, mass production was done both for AFS composite with pure Zn as brazing joint material and AFS composite with Zn2%Al as brazing joint material.

As following, solution heat treatment was conducted to the half of the produced AFS composite and investigated with characterization methods which they were 3 point bending, SEM, EDS and micro hardness test. All the results were explained on the results and discussion section.

### **3.2.1 Preparation Works of Production AFS Composites**

There are some sequences of preparation works for samples and equipment which were used during the production of AFS composites and they consist on calibration and cleaning of the furnace, cutting operation of the sandwich components, soft polishing sandwich component surfaces in order to eliminate the oxidation layers.

And as following, putting Al-foam inside the nitric acid 12% solution for cleaning, ultrasonic cleaning by Sonica for all the sandwich components, application of the Al-flux on the surface of Al-7046 components and positioning of sandwich components and adding additional components before putting inside the furnace.

In this section, all the preparation methods were explained for my experiments of production AFS composites.

### **3.2.1.1 Grinding**

As following to cutting operation, it was needed to prepare the samples for joining activity. However, all the AFS components have got oxide layers on the surfaces due to air that there is free oxygen inside the air and they were oxidized.

Oxide removing operation was done for all the AFS components by hand with polishing plate. Abrasive polishing plate number 320 was performed until seeing the oxide layer was removed for Al-7046 sheets and pure Zn or Zn2%Al. After removing the thin layer of oxide on the surface, Al-7046 sheets and pure Zn or Zn2%Al plates were seen brighter than the first look.

Also short time soft polishing was done for Al-foam because of it is essential to have flat surface instead of sharp curves on the surface. This is really important because the AFS composite structure has got many layers; the first one Al plate then pure Zn or Zn2%Al then Al-foam then again Zn or Zn2%Al and Al plate.

The joining of these different layers was done by the melt of Zn or Zn2%Al components on the Al-foam and Al plate. For that reason, if the Al-foam has got sharp edges on the surface, this melt would not find enough surface area in order to obtain joining between Al-foam and Al plate. For that reason, it is really essential to have flat surface on Al-foam in order to increase the flat surface area which has got connection with melt for joining.

During soft polishing by hand, some abrasive particles from polishing plate and oxide layer on the surface can be emerged to the air. It is hazardous to inhale these particles thus, it is recommended using mask and working under hood with the ventilation system on.

### **3.2.1.2 Nitric Acid 12% Solution**

Following step is putting Al-foams inside the nitric acid 12% solution for 10 minutes. This step was done for the reason of Al-foam pore structure that they have open pores inside the structure and it is important to clean these areas from unknown particles. If the purifying cleaning was not done, some problems on Al-foam might occur during the brazing joining treatment due to high temperature.

Nitric acid 70% was used for cleaning pore structure of Al-foams however it was purified with distilled water in an amount that having the solution 12% of Nitric acid. This step is dangerous due to usage of acid. Special laboratory gloves and uniform must be wear and must be working under the hood with ventilation system is on during this operation. Also, the Al-foams should be cleaned with distilled water after removing them out from the 12% nitric acid solution.

### 3.2.1.3 Ultrasonic Cleaner

When the Al-foams were removed from nitric acid solution and the oxide layers were eliminated from the surface of the samples, another cleaning operation was implemented for all the AFS components so called ultrasonic cleaning. This operation was done at temperature 60 °C for 10 minutes by the Sonica ultrasonic cleaner, see figure 44. Firstly, all the components were put inside the laboratory glass which filled with pure alcohol then this glass was placed inside the ultrasonic cleaner.



Figure 44: Ultrasonic cleaner

Ultrasonic cleaning is the rapid and complete removal of contaminants from objects which are immersed in a tank of liquid that is flooded with high frequency sounds waves. These non-audible sound waves create a gentle scrubbing action within the fluid, removing contaminants from all surface areas the fluid comes into contact with [25].

The process is brought about by high frequency electrical energy that is converted by a transducer into high frequency sound waves, ultrasonic energy. Ultrasonic energy enters the liquid within the tank or bath this causes the rapid formation and collapse of minute bubbles; a phenomenon known as cavitation. These bubbles travel at high speed within the tank, causing them to implode against the surface of any immersed object with an enormous release of energy. As the bubbles implode and cavitation occurs, the cleaning solution rushes into the gap left behind by the bubbles, gently lifting any contaminants, dirt and tarnishing from both the surface and innermost recesses of intricately shaped parts [25].

### 3.2.1.4 Application of Al Flux

When all the cleaning operations were done for AFS components, Al flux Al6 was applied on the inner surfaces of the Al-7046 sheets which touch the pure Zn or Zn2%Al sheet inside the AFS composite. Al6 flux was recommended for brazing with Zn-Al filler metals. Before the application, Al flux always must be mixed well.

Al6 flux contains cesium fluoroaluminate and it should be avoided to contact with skin, eyes thus using laboratory gloves and protector equipments were essential. The main reason to use flux was they behave as deoxidizers and preventing re-oxidation of aluminium sheet.

### 3.2.2 Trials for Finding Optimum Furnace Parameters

In order to find the optimum furnace parameters for brazing joining treatment, some experiments of producing one side AFS composite were done. Firstly, it was started with the assumed parameters which applied for the 1<sup>st</sup> batch of Al-7046. Then results of the joining were investigated and new parameters were decided until obtaining the optimum strong joint between Al sheet and Al-foam.

All the furnace operations for brazing joining treatment were done in Argon gas atmosphere. Furnace cleaning was done again every time before using the furnace. After placing the samples in a correct location inside the furnace tube, furnace was turned on and parameters of the treatment were entered on the furnace screen. Firstly, the brazing temperature then dwell time, heating and cooling rates.

Then subsequently, water and Argon gas turned on. To understand the Argon gas is working, the hose of the Argon gas was put inside the glass filled by water, shown on the figure 45, also Argon gas green tubes behind the furnace. When there were some bubbles inside the water, it means that gas is on.

Water supply was on also in the hose for circulation on the head of the furnace tube in order not to have overheating. It should be always considered that the plug on the furnace entrance might remove out due to pressure of some gasses inside the tube and the Argon gas might be stopped during the treatment. For these reasons, it is always better to check the furnace with short time intervals.

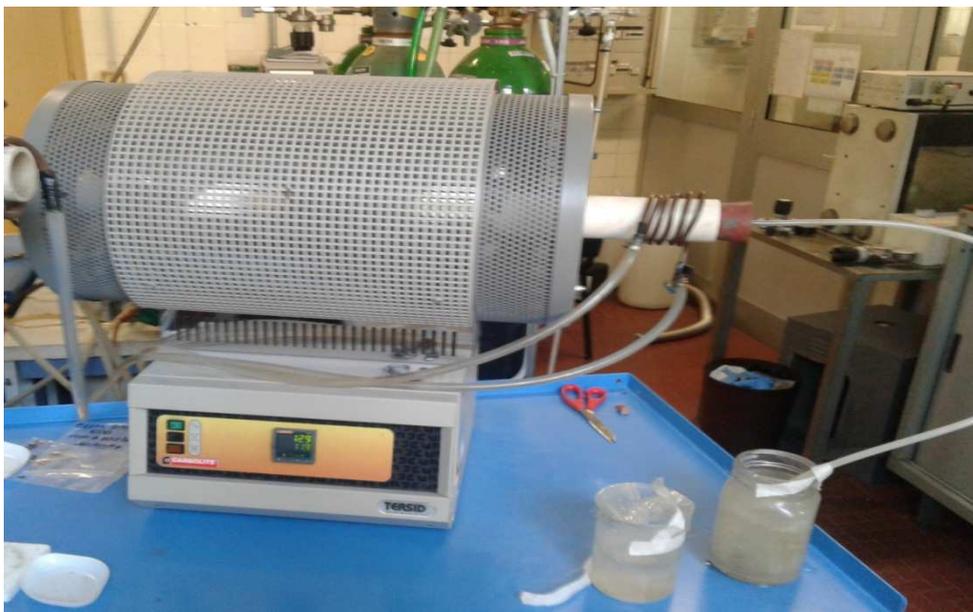


Figure 45: Brazing joining heat treatment in the furnace

### 3.2.2.1 First Experiment

The first assumption of the brazing furnace parameters for producing AFS composite were brazing temperature 450°C, dwell time at maximum temperature for 10 minutes, heating rate 16.6°C per minute and cooling rate 10 °C per minute.

The calibration of the furnace was already done before the trials and furnace calibration table was obtained. When it was checked the corresponding temperature for 450°C, it was seen 553°C as corresponding temperature. So, while furnace parameters were entering to the screen, 553°C was entered as brazing temperature which means it was real 450°C temperature inside the furnace tube.

All the process of heating, dwell and cooling took around 4 hours. When the cooling was over, subsequently furnace heating, furnace water, water supply and Argon gas was turned off. Then furnace entrance plug was removed and AFS composite was removed out by metal furnace stick. The result of the first experiment was failure as shown in the figure 46 below. The joint between Al sheet and Al-foam was not obtained and all the components were separated. Zinc was not melted properly and there was not enough diffusion between the layers.

In order to melt the Zinc component, the decision was taken to increase the brazing temperature.



Figure 46: 1<sup>st</sup> experiment

### 3.2.2.2 Second Experiment

The brazing temperature was increased to 470°C from 450°C in the second experiment. It was conducted with the same heating, cooling rates and dwell time at maximum temperature. The corresponding temperature for 470°C was checked on the calibration table and it was 571°C.

Thus, while entering the parameters of the second experiment on the furnace screen, 571°C was entered for brazing temperature which means that the real temperature inside the furnace tube was 470°C. The same furnace procedures were performed, the same like first experiment for starting up the furnace experiment and ending it up. The result was the similar like the first experiment, joint was not obtained again due to Zinc was not melted properly. For melting the Zinc component, it was needed to go higher brazing temperatures. So, the decision was taken again to increase to brazing temperature from 470°C.

### 3.2.2.3 Third Experiment

For the third experiment, it was decided to increase the brazing temperature and it was increased to 500°C from 470°C. Heating, cooling rates and dwell time at maximum temperature were the same like previous 2 experiments. The corresponding temperature of 500°C was checked on the calibration table and it was 599°C thus this temperature was entered to the furnace screen during setting up which means that the real temperature inside the furnace tube was 500°C.

The same furnace procedures were performed for starting up the furnace experiment and ending it up. When the AFS composite was removed out from the furnace, it was checked and the result was good. It was obtained good strong joint between Al sheet and Al-foam. Then the small sized 3 cm to 2 cm AFS composite was cut from the middle by cutting machine in order to check the joint again and the result was also good, it is shown in the figure 47.

As final, it was decided to have brazing temperature 500°C for mass production of the real sized AFS composites. The other parameters for heating, cooling rates and dwell time at maximum temperature were the same like previous experiments.



Figure 47: 3<sup>rd</sup> experiment AFS composite which is cut from the middle

### 3.2.3 Production of AFS Composite with Pure Zinc

Previously on experiments, optimum furnace parameters were obtained for producing the small sized AFS composite; brazing temperature 500°C, heating rate 16.6°C per minute, cooling rate 10°C per minute and 10 minutes dwell time at maximum temperature. It was needed to apply these furnace parameters in order to produce bigger size of AFS composites for 3 point bending test which the dimensions were 60 mm to 20 mm.

For 3 point bending test, it was necessary to produce 6 pieces AFS composites with brazing joining treatment using pure Zn as brazing joining material in Argon gas atmosphere and following to brazing joining heat treatment, applying to 3 pieces AFS composites solution heat treatment which that treatment helps us to increase mechanical properties such as hardness, strength after brazing heat treatment. It was seen the positive effect of doing solution heat treatment for the 1<sup>st</sup> batch of Al-7046 plates, kind of recovery mechanical properties back after brazing heat treatment.

The aim was at the end, obtaining 3 pieces of AFS composites without solution heat treatment and 3 pieces of AFS composites with solution heat treatment. After producing 3 pieces AFS composite without solution heat treatment and 3 pieces AFS composite with solution heat treatment, I could conduct some characterization methods in order to investigate the importance and influence of the solution heat treatment on the AFS composites if they were really necessary to apply in order to increase their properties.

All the preparation of the AFS components were the same before, cutting was obtained by designated cutting machine for Al-7046 plates and Al-foams with the dimension 60mm to 20mm and pure Zn was cut by scissors.

### 3.2.3.1 Brazing (Joining) Heat Treatment

Brazing joining heat treatment was applied to the AFS components in the same furnace, Carbolite Tersid. As mentioned before, it was needed to produce 6 pieces of AFS composites with pure Zn as joining material and it was started to produce AFS composites with the optimum parameters which were obtained before.

#### 3.2.3.1.1 First Experiment

The furnace parameters of the first experiment for producing AFS composites were, brazing temperature 500°C, heating rate 16.6°C per minute, cooling rate 10°C per minute and dwell time 10 minutes at the maximum temperature, in argon atmosphere.

When the results of the first experiment were checked, brazing and joint between Al sheet and Al-foam was good and strong however it was witnessed that over melting and gas holes were shown on the one surface of AFS composite, see the figure 48.



Figure 48: First experiment, AFS

These problems were might happened due to heat transfer between Al plate and graphite base profile during the long dwell time at the maximum temperature. So, it was needed to decrease to dwell time from 10 minutes and decision was taken to make dwell time for 5 minutes.

### 3.2.3.1.2 Second Experiment

In order to decrease the melting area and gas holes on the surface, dwell time was decreased and the furnace parameters of the second experiment for producing AFS composites were, brazing temperature 500°C, heating rate 16.6°C per minute, cooling rate 10°C per minute and dwell time 5 minutes at the maximum temperature, in argon atmosphere. When the results of the second experiment were checked, brazing and joint between Al sheet and Al-foam was good and strong again. Comparing the first experiment, almost no over melting on the surface and less gas holes was obtained on the surface, see the figure 49. Furnace parameters were good and okay for production of the AFS composites. However, trying to find the optimum parameters in order not to have any gas holes and any over melting on the surface, it was decided to decrease the brazing temperature so another experiment was conducted with the lower brazing temperature.



Figure 49: Second experiment, AFS

### 3.2.3.1.3 Third Experiment

With the decrease of the brazing temperature, the new furnace parameters of the third experiment for producing AFS composites were, brazing temperature 490°C, heating rate 16.6°C per minute, cooling rate 10°C per minute and dwell time 5 minutes at the maximum temperature, in argon atmosphere. When the results of the third experiment were checked, brazing and joint between Al sheet and Al-foam was failed. The joint between Al sheet and Al-foam was not obtained on the one side of the AFS composite, see the figure 50.



Figure 50: Third experiment, AFS

It was understood from the result that decreasing brazing temperature from 500°C was not good solution because of the joint between Al sheet and Al-foam was not obtained. So, the lower limit of the brazing temperature for having joint was 500°C and it was not possible to modify it. Then the new decision was taken to decrease the dwell time a bit more in order to investigate new results.

### 3.2.3.1.4 Forth Experiment

With the decrease of the dwell time, the new furnace parameters of the forth experiment for producing AFS composites were, brazing temperature 500°C, heating rate 16.6°C per minute, cooling rate 10°C per minute and dwell time 2.5 minutes at the maximum temperature, in argon atmosphere. When the results of the forth experiment were checked, the joint between Al sheet and Al-foam was obtained.

### 3.2.3.1.5 Finding Optimum Experiment for Production

Comparing all these 4 experiments in order to find the optimum experiment for mass production, experiment 3 was already failed due to low brazing temperature, experiment 1 had over melting surface and experiment 4 had less time for dwell which might cause less strength on the joint.

As final, the optimum experiment was selected number 2 for mass production of AFS composites and the brazing parameters as following; brazing temperature 500°C, heating rate 16.6°C per minute, cooling rate 10°C per minute and dwell time 5 minutes at the maximum temperature, in argon atmosphere. Brazing treatment parameters for producing AFS composites with pure Zn as brazing joint material are reported on the diagram in figure 51.

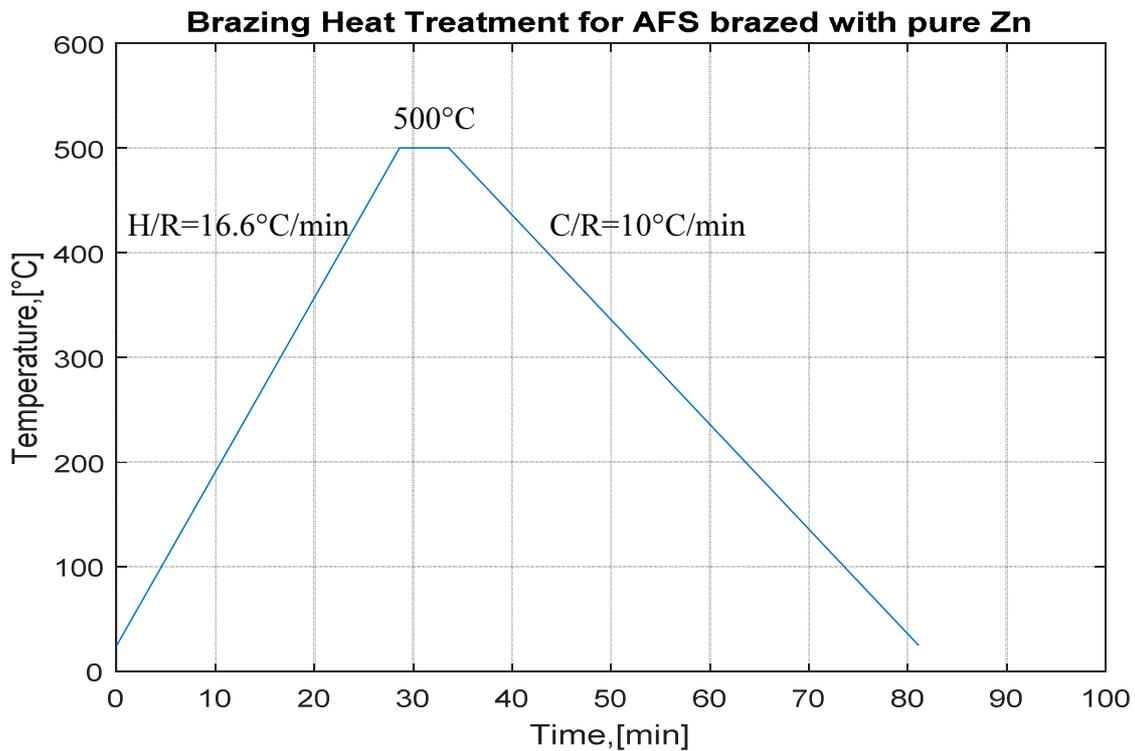


Figure 51: Optimum brazing heat treatment for AFS brazed with pure Zn

Accordingly to the diagram above, 6 pieces of AFS composites with pure Zn as brazing joint material were produced.

### 3.2.3.2 Solution Heat Treatment

As following to the production of 6 pieces of AFS composites with pure Zn as brazing joint, solution heat treatment was conducted for 3 pieces of AFS composites.

Solution heat treatment was conducted with the parameters; temperature 370°C, heating rate 12°C per minute, dwell time at maximum temperature for 3 hours and water quench, in Ovmat 2009 furnace, reported in the diagram in figure 52.

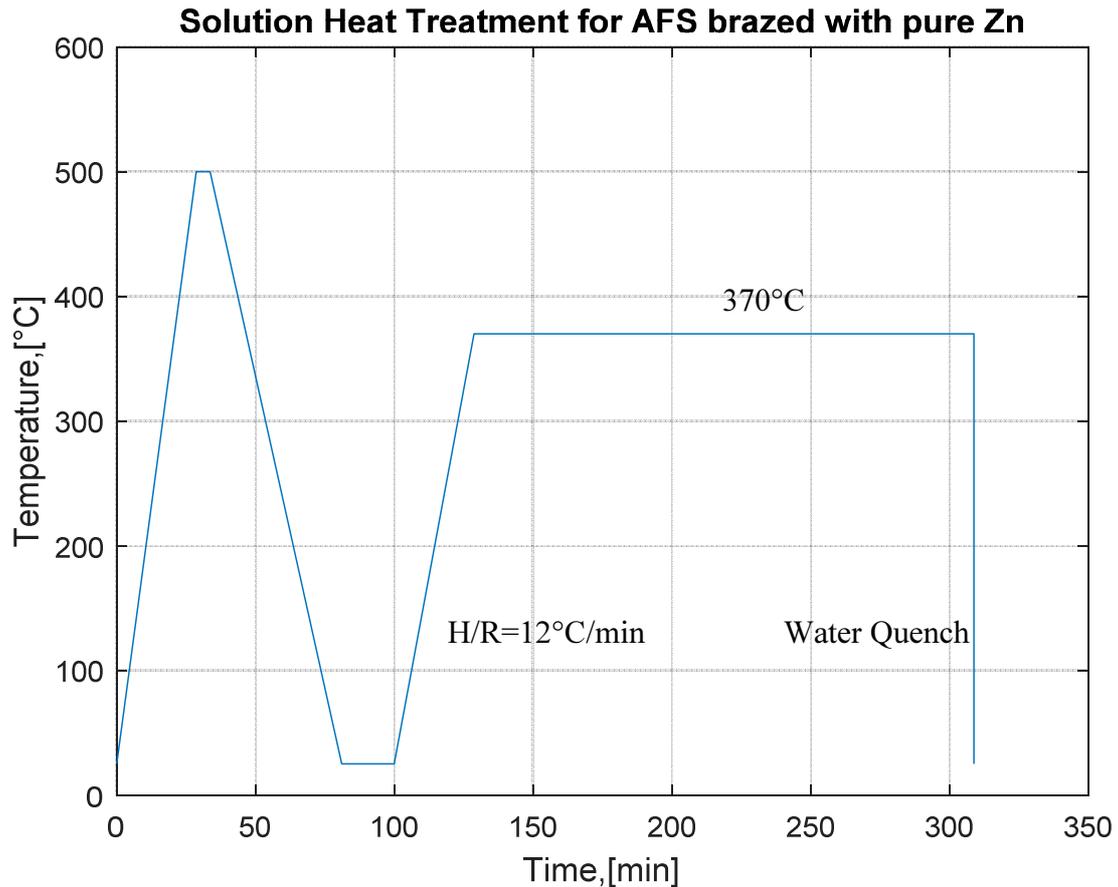


Figure 52: Solution heat treatment for AFS brazed with pure Zn

### 3.2.4 Production of AFS composite with Zn2%Al

Production of the AFS composite with Zn2%Al as brazing joint material was the same like production of AFS composite with pure Zinc. All the preparations, cleanings and furnace processes were the same. Just the difference was using Zn2%Al instead of using pure Zn as brazing joint material inside the AFS composite for having joint between Al sheet and Al-foam. The aim was to produce 6 pieces of AFS composite with Zn2%Al. After producing 6 pieces, solution heat treatment was performed on 3 of them.

### 3.2.4.1 Brazing (Joining) Heat Treatment

6 pieces AFS composite with Zn2%Al as brazing joint material were produced with brazing joining treatment with the following parameters;

Brazing temperature 520°C, heating rate 16.6°C per minute, cooling rate 10°C per minute and dwell time at maximum temperature for 5 minutes in Argon gas atmosphere with the same brazing furnace Carbolite Tersid, reported in figure 53.

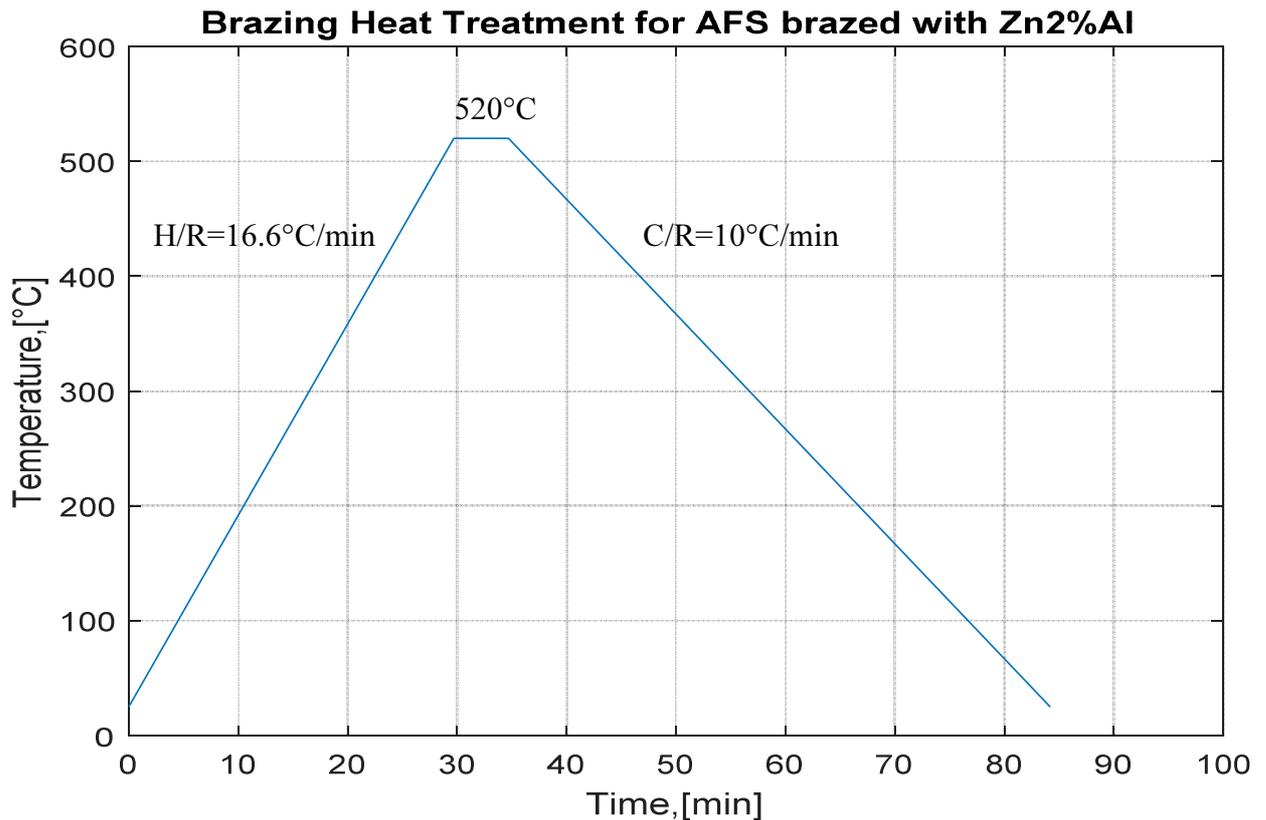


Figure 53: Brazing heat treatment for AFS brazed with Zn2%Al

The result of the production of AFS composite with Zn2%Al is shown in the figure 54, both brazing and joining between Al plate and Al-foam was good. Then 5 more pieces of AFS composite were produced with the parameters above. At final, 6 pieces of AFS composite were obtained.



Figure 54: Result of the brazing parameters, AFS composite brazed with Zn2%Al

### 3.2.4.2 Solution Heat Treatment

After producing 6 pieces of AFS composites brazed with Zn2%Al, as following solution heat treatment was conducted for 3 pieces of AFS composites in the same solution heat treatment furnace, Ovmat 2009, with the following parameters;

Solution heat treatment temperature 370°C, heating rate 12°C per minute, dwell time at maximum temperate for 3 hours and water quench, shown in the figure 55.

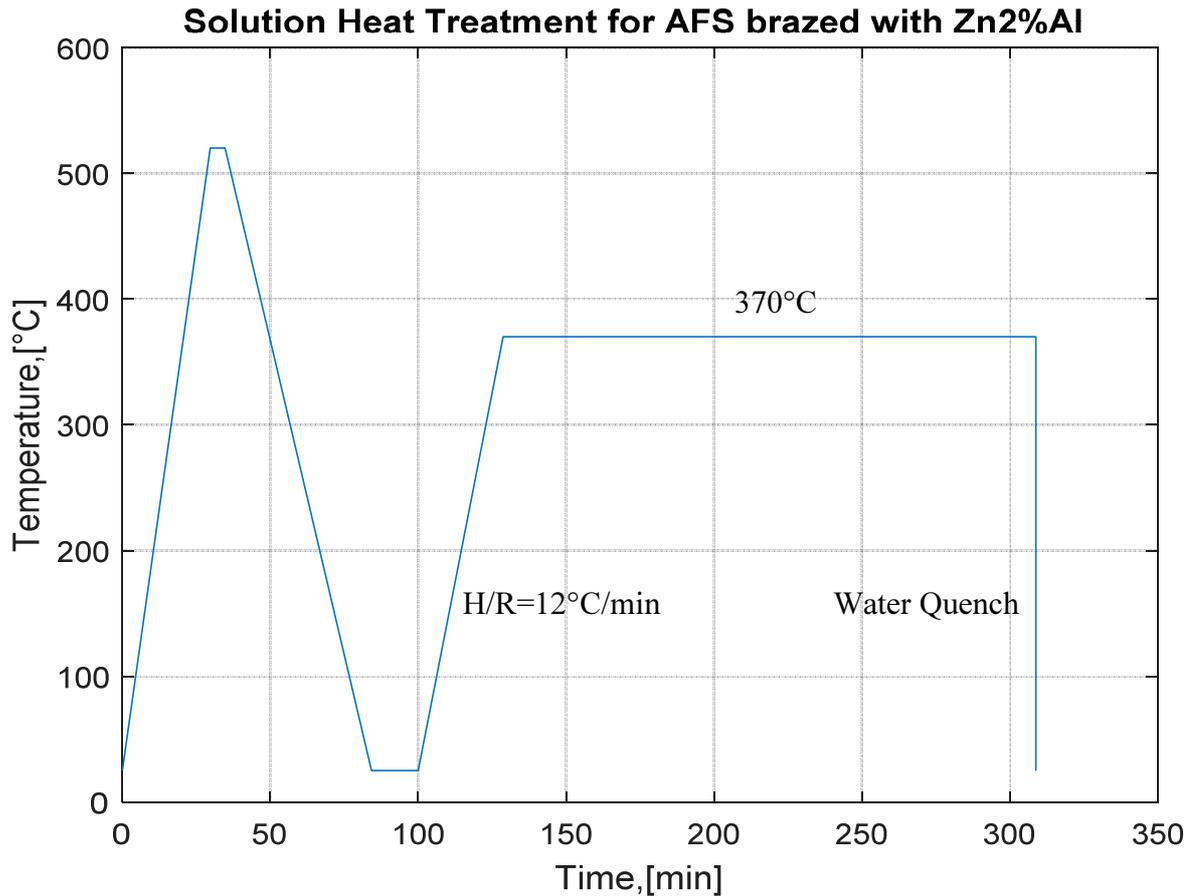


Figure 55: Solution heat treatment for AFS brazed with Zn2%Al

After the solution heat treatment which applied to 3 pieces of AFS brazed with Zn2%Al, production of AFS composites were over.

The total AFS composites production, I obtained 3 pieces AFS brazed with pure Zn, 3 pieces solution heat treated AFS brazed with pure Zn, 3 pieces AFS brazed with Zn2%Al and 3 pieces solution heat treated AFS brazed with Zn2%Al.

### 3.2.5 Characterization Methods for AFS Composite

There are some characterization methods subsequently were conducted both for the AFS composites brazed with pure Zn and brazed with Zn2%Al also both for solution heat treated and non solution heat treated AFS composites.

The characterization methods were as following; 3-point bending test, scanning electron microscopy (SEM), energy dispersive spectrometry (EDS) and micro hardness test.

#### 3.2.5.1 3-Point Bending Test

3-point bending test were performed for in total 12-pieces of AFS composites with dimension 60mm to 20mm, see figure 56.

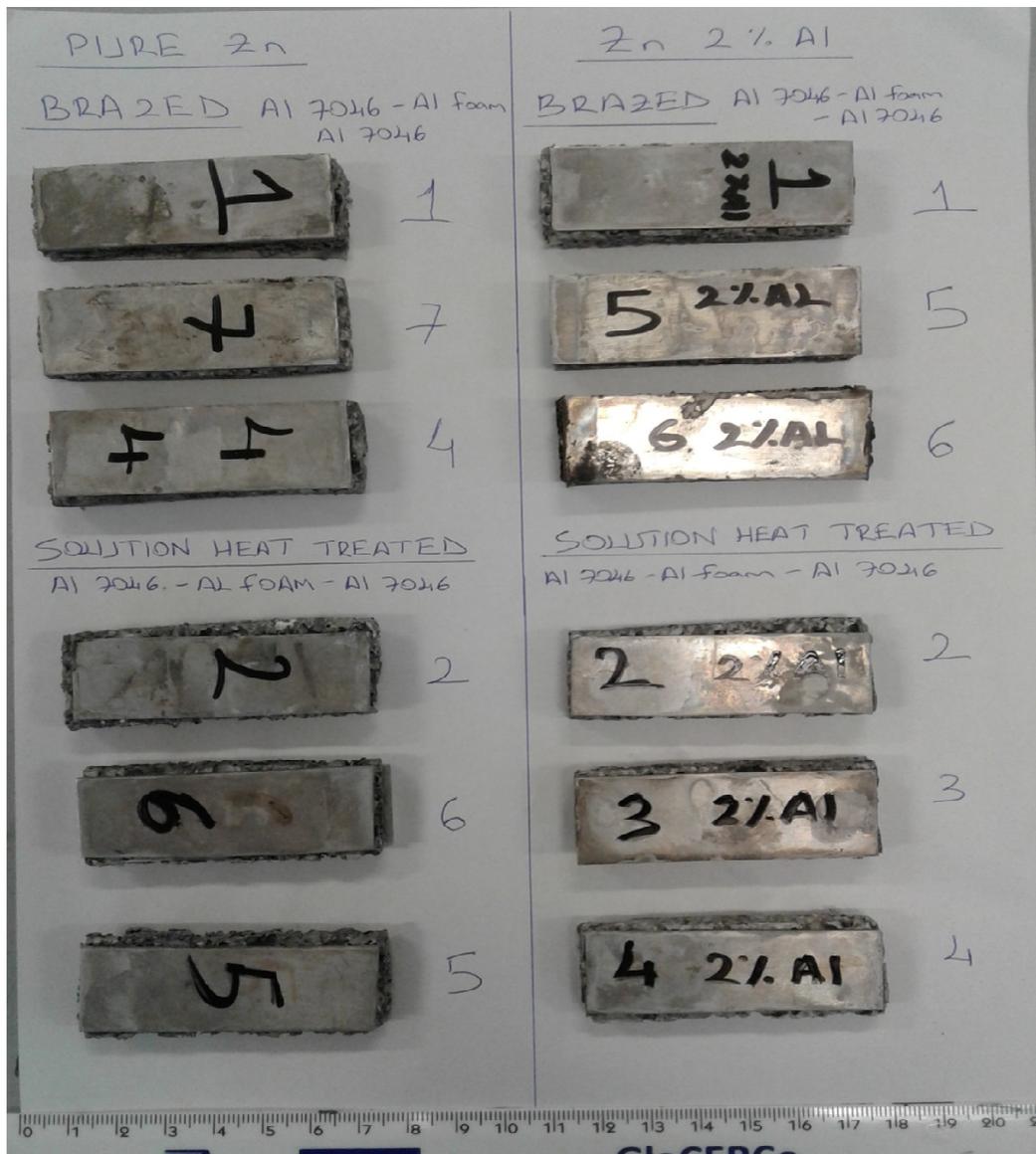


Figure 56: All AFS composites before 3-point bending test

These 12 pieces AFS composites consisted on;

- 3-pieces AFS brazed with pure Zn (on the left), 3-pieces solution heat treated AFS brazed with pure Zn (on the right) in figure 57 below.



Figure 57: AFS composites with pure Zn before 3 point bending test

- 3-pieces AFS brazed with Zn2%Al (on the left), 3-pieces solution heat treated AFS brazed with Zn2%Al (on the right) in figure 58 below.

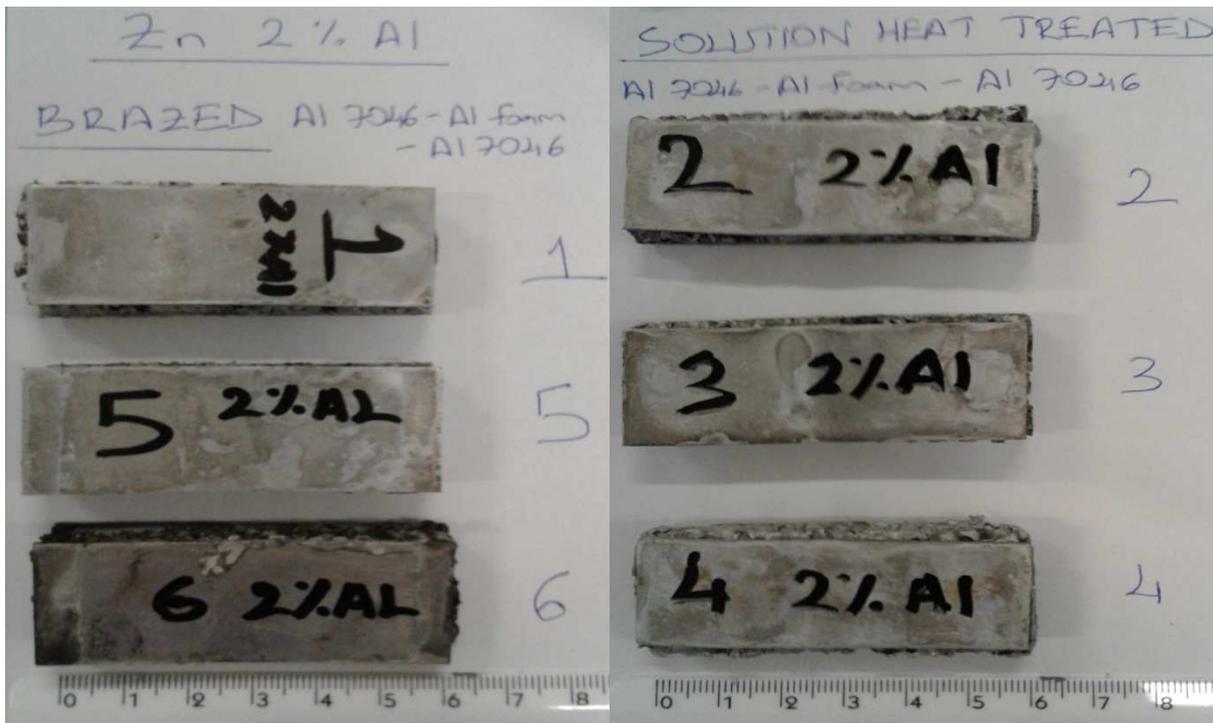


Figure 58: AFS composites with Zn2%Al before 3 point bending test

Number 1 AFS composite brazed with pure Zn, beginning on the test (on the left) and end of the test (on the right) are shown in the figure 59. End of the test, crack was happened on the down side of Al-7046 plate.

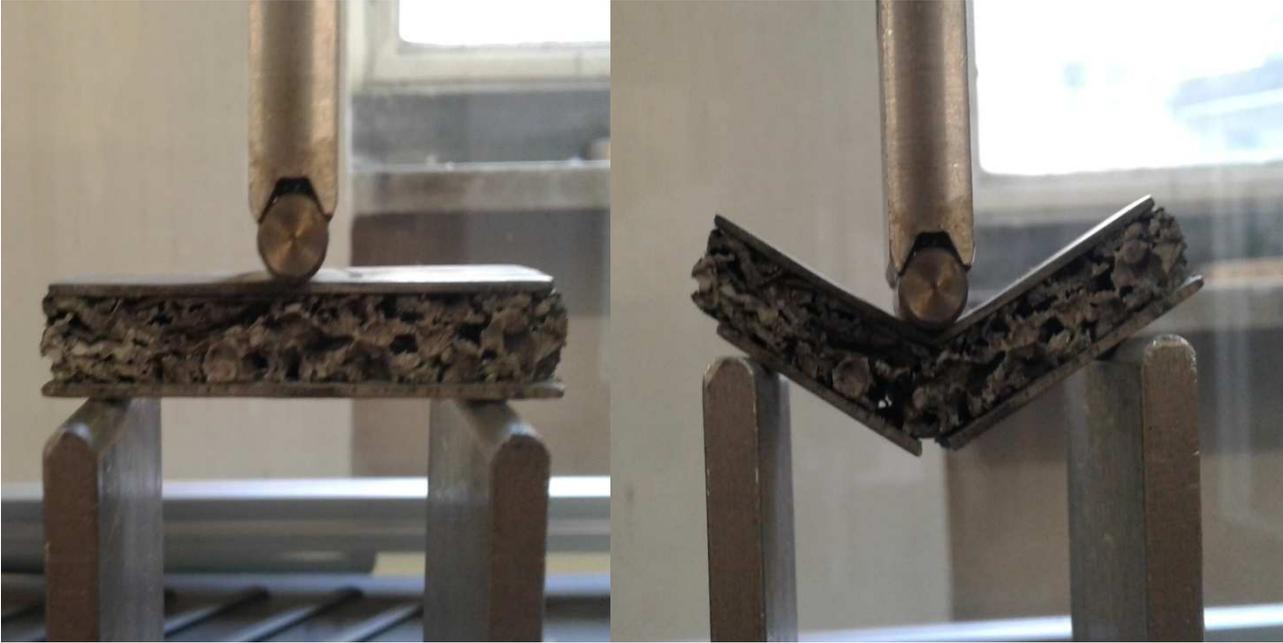


Figure 59: 3 point bending test for number 1 AFS composite

Also, when the 3 point test was performed for the number 7 AFS composite brazed with pure Zn, beginning of the test (on the left) and end of the test (on the right) are shown in the figure 60. Crack was not happened at this time.

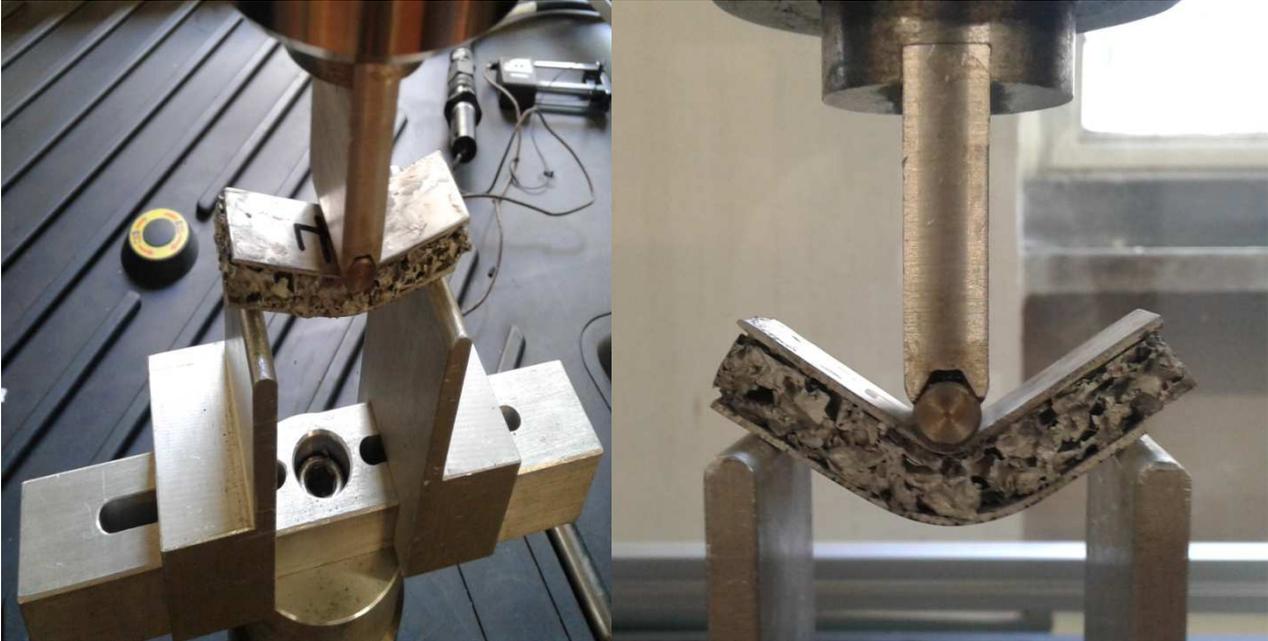


Figure 60: 3 point bending test for number 7 AFS composite

End of the 3 point bending tests, some cracks or separations of the joint between Al plate and Al-foam or cracks and no separation were obtained on the AFS composites. The results of the appearance of the AFS composites after 3 point bending are shown as following;

- 3-pieces AFS brazed with pure Zn (on the left), 3-pieces solution heat treated AFS brazed with pure Zn (on the right) after 3 point bending test in figure 61.



Figure 61: AFS composites with pure Zn after 3 point bending test

- 3-pieces AFS brazed with Zn2%Al (on the left), 3-pieces solution heat treated AFS brazed with Zn2%Al (on the right) after 3 point bending test in figure 62.

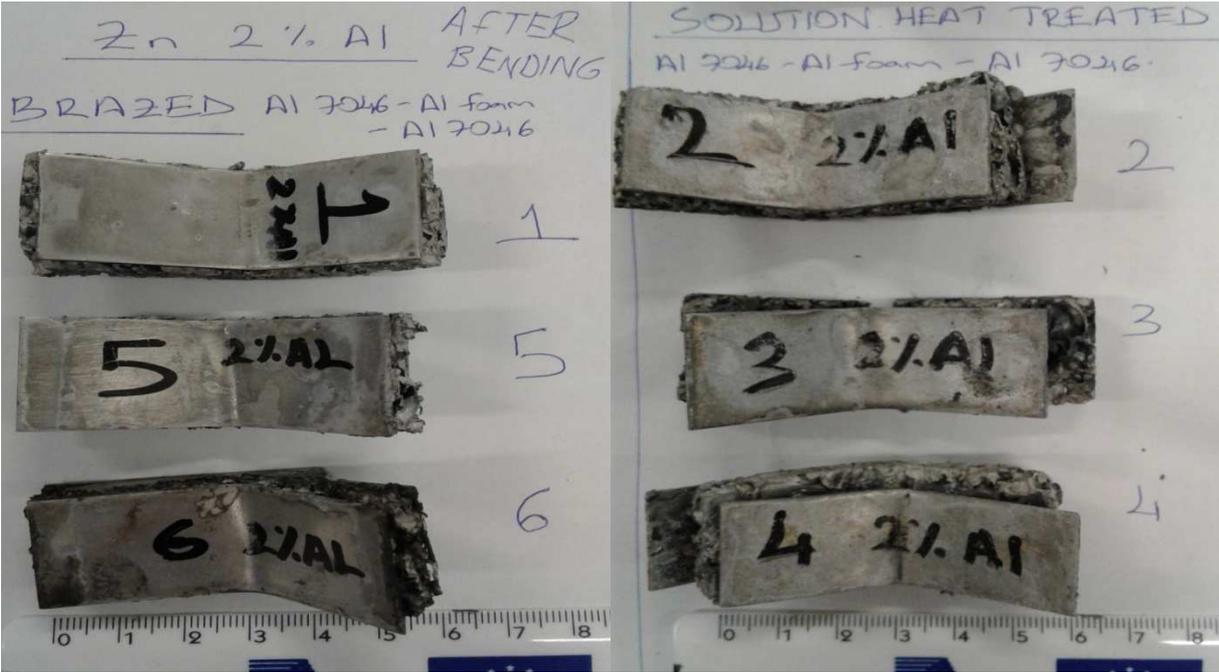


Figure 62: AFS composites with Zn2%Al after 3 point bending test

### **3.2.5.2. Scanning Electron Microscopy (SEM)**

As following to the bending test of the AFS composites, scanning electron microscopy was performed to investigate the joint structure, Al-foam structure and Al plates both for pure Zn as brazing joint material and Zn2%Al as brazing joint material and both for solution heat treated and non solution heat treated AFS composites.

The same preparation methods were done briefly as following; cutting the AFS composites into small samples with designated cutting machine then gloss coating with epoxy resin and hardener. After around 10 hours, when the samples were hardened, polishing operation on the surfaces in order to have bright smooth skin.

Next step, before the scanning electron microscopy, was assembly the metal SEM tools on the base of the gloss coated sample and application of silver liquid from the top surface to the base until reaching the metal SEM tools. Then just before the scanning electron microscopy, putting samples inside the low temperature furnace in order to eliminate all the water particles and water drops inside the Al-foam and on the surface.

### **3.2.5.3 Energy Dispersive Spectrometry (EDS)**

The Zeiss Merlin machine has got the functions of making EDS and SEM analyzes together due to X-ray spectrometer is added on it. After achieving electron images with SEM, it is also possible to have element mapping, even position analysis and area analysis at the same time. These 2 functions were together in one instrument.

When the desired electron image is obtained for a point or an area, it is possible to have the chemical analysis for seeing all the particular elements and their relative proportions on selected point. With EDS analysis, I obtained the chemical structures of AFS composites such as; AFS brazed with pure Zn, solution heat treated AFS brazed with pure Zn, AFS brazed with Zn2%Al and solution heat treated AFS brazed with Zn2%Al. When I had the results of the composition of the elements on the joint area, on the plate and on the Al-foam, I could make my investigation about the effects of the heat treatments and effects of the brazing joining materials in terms of chemical structures.

### **3.2.5.4 Micro Hardness Test**

Vickers micro-hardness test was performed subsequently to SEM and EDS analyzes. The aim of this hardness test was to understand the effect of conducting different heat treatments and the effect of using different brazing joint materials on the hardness of the samples. For Vickers micro-hardness test, I used again the HX-1000 Remet hardness instrument which is directly connected to the computer. The selected operation parameters were dwelling time 15 seconds and force 25gf. Vickers micro-hardness tests were conducted to the joint areas (diffusion areas) between Al-foam and Al-7046 plate, Al-foams and Al plates of AFS composites brazed with pure Zn, solution heat treated AFS composites brazed with pure Zn, AFS composites brazed with Zn2%Al and solution heat treated AFS composites brazed with Zn2%Al.

### **3.3 2nd Batch of Al-7046 Alloys**

In order to investigate the effects such as hardness, strength, elongation, microstructure and chemical structure, of the brazing heat treatment and solution heat treatment on the Al-7046 plates, 2<sup>nd</sup> batch of the Al-7046 alloys were produced with braze joining heat treatment conditions which the same parameters of brazing AFS components.

And, solution heat treated Al-7046 alloys were produced with solution heat treatment conditions which the same parameters of solution heat treatment to the AFS composites.

While producing AFS composites with braze joining heat treatment, at the same time, also 6 pieces of as received Al-7046 sheets with the dimension 60mm to 20mm and two small pieces of as received Al-7046 were put inside the brazing furnace.

Following to the brazing process, 3 of these brazed pieces and one small brazed piece were put inside the solution heat treatment furnace while obtaining solution heat treatment for AFS composites at the same time.

In the final of these processes, I obtained 3 pieces of brazed Al-7046, 3 pieces of solution heat treated Al-7046 with the dimensions 60mm to 20mm for 3 point bending test. Also, one small piece of brazed Al-7046 and one small piece of solution heat treated Al-7046.

These small pieces, brazed, solution heat treated and as received Al-7046, were cut to the dimension 10mm to 10mm for gloss coating then subsequently some characterisation methods were done the same like previously done for 1<sup>st</sup> batch of Al-7046 alloys such as optical microscopy, SEM, EDS and micro hardness tests.

#### **3.3.1 Brazing Heat Treatment**

Brazing heat treatments for 6 pieces dimensions 60mm to 20mm and 2 small pieces of as received Al-7046 as 2<sup>nd</sup> batch were conducted by designated brazing furnace with the same parameters of brazing AFS components as following; brazing temperature 500°C, heating rate 16.6°C per minute, cooling rate 10°C per minute and dwell time at maximum temperature for 5 minutes.

The brazing parameters for 2<sup>nd</sup> batch of Al-7046 plates are shown in following figure 63.

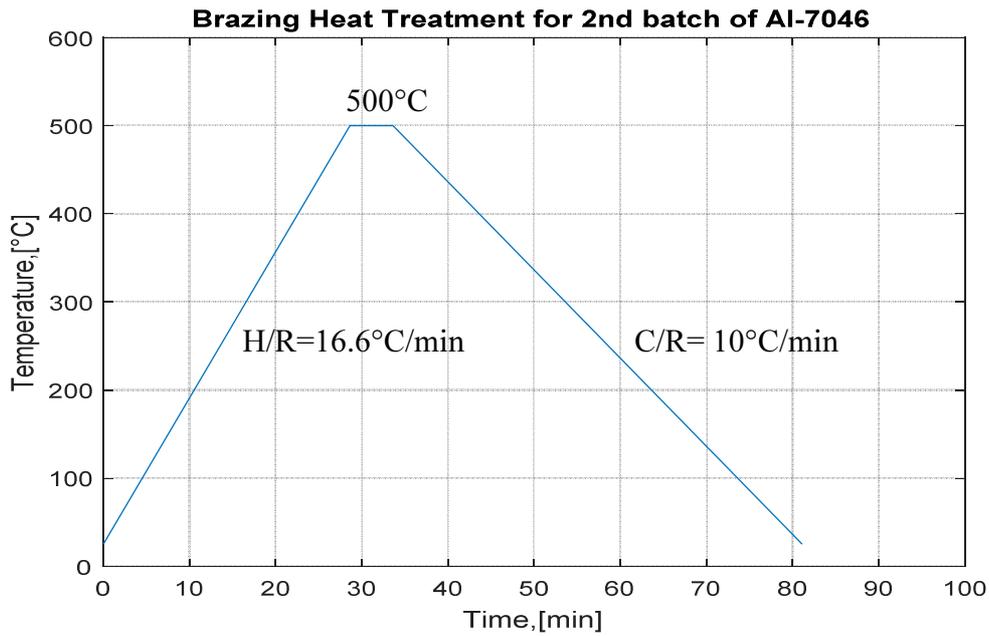


Figure 63: Brazing heat treatment for 2<sup>nd</sup> batch of Al-7046

### 3.3.2 Solution Heat Treatment

Solution heat treatments for 3 pieces dimensions 60mm to 20mm and 1 small piece of brazed heat treated Al-7046 as 2<sup>nd</sup> batch were conducted by designated solution heat treatment furnace with the same parameters of solution heat treatment for AFS components as following; temperature 370°C, heating rate 12°C per minute, dwell time at maximum temperature for 3 hours and water quench. The solution heat treatment parameters for 2<sup>nd</sup> batch of Al-7046 plates are shown in the figure 64.

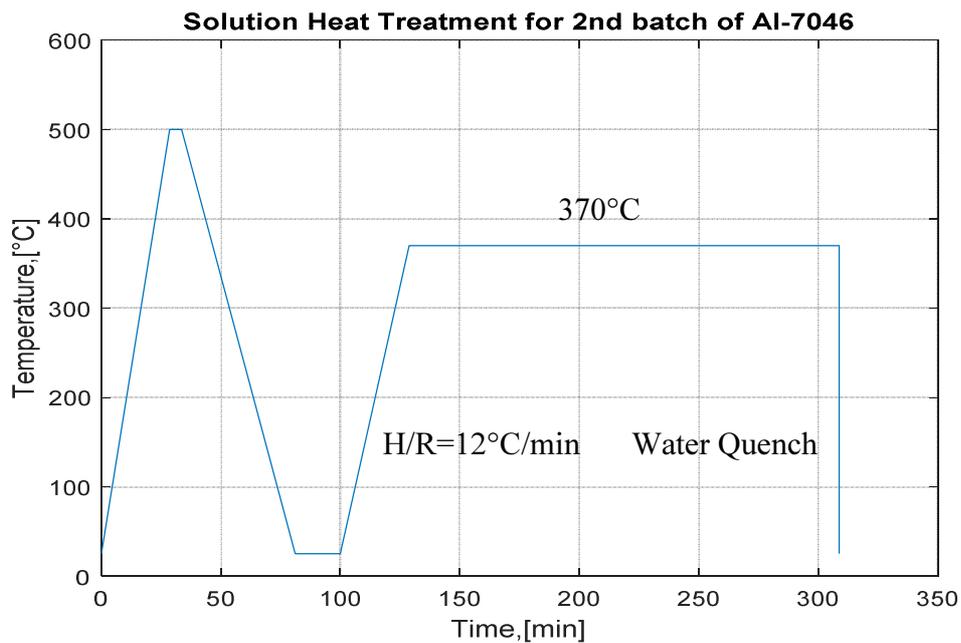


Figure 64: Solution heat treatment for 2<sup>nd</sup> batch of Al-7046

### 3.3.3 Characterization Methods

After obtaining 3 pieces of brazed Al-7046 and 3 pieces of solution heat treated brazed Al-7046 with the dimensions 60mm to 20mm, they were ready to conduct 3 point bending test with the same bending machine explained previously. In order to compare the mechanical properties between Al-7046 plates and AFS composites, it was essential to obtain 3 point bending test also for Al-7046 plate without sandwich construction and foam.

All the 3 point bending samples before (on the left) and after (on the right) the test are presented in the following figure 65, AR represents for as received and there were 3 samples from each group, B represents for brazed heat treated and SH represents for solution heat treated. The results of the bending tests were published and explained on the results chapter.



Figure 65: 3 point bending test for 2<sup>nd</sup> batch of Al-7046, left before, right after

On the other side, produced one small piece of brazed and solution heat treated Al-7046 were prepared for optical microscopy, scanning electron microscopy (SEM), energy dispersive spectrometry (EDS) and micro hardness test. The same procedures were applied also for 2<sup>nd</sup> batch of Al-7046, subsequently, cutting operation of the samples to dimension 10mm to 10mm after gloss coating with epoxy resin and hardener then polishing the samples. When the polished gloss coated samples were obtained, optical microscopy, SEM, EDS and micro hardness tests were conducted.

After producing 2<sup>nd</sup> batch of Al-7046 alloys and completing their characterization methods, all the productions and tests were over. Now, it is time to publish and discuss the results of the characterization tests for 1<sup>st</sup> batch and 2<sup>nd</sup> batch Al-7046 alloys, brazed and solution heat treated AFS composites both brazed with pure Zn and Zn2%Al.

# CHAPTER 4.RESULTS AND DISCUSSION

In this chapter, all the characterization results of optical microscopy, SEM, EDS, micro hardness and 3 point bending test were published and discussed for 1<sup>st</sup> and 2<sup>nd</sup> batch of Al-7046 Alloys, AFS composites brazed with pure Zn and Zn2%Al both for solution heat treated and non solution heat treated, also, micro hardness of the Al-foam for different heat treatments.

## 4.1 1st Batch of Al-7046 Alloys

1<sup>st</sup> batch of Al-7046 alloys consist of as received, brazed heat treated and solution heat treated Al-7046 alloys.

### 4.1.1 Optical Microscopy

Images were obtained with the different objective lens and eyepiece magnifications such as 100x-1.88x, 100x-1.25x and 50x-1.25x for as received (AR), brazed heat treated (B) and solution heat treated (SH) Al-7046 alloys.

- **100x-1.88x Magnification**



Figure 66: Microstructures as received (AR-left), brazed heat treated (B-middle), solution heat treated (SH-right) with 100x-1.88x magnification

- **100x-1.25x Magnification**



Figure 67: Microstructures as received (AR-left), brazed heat treated (B-middle), solution heat treated (SH-right) with 100x-1.25x magnification

- **50x-1.25x Magnification**



Figure 68: Microstructures as received (AR-left), brazed heat treated (B-middle), solution heat treated (SH-right) with 50x-1.25x magnification

After obtaining the images from the optical microscope, I am not able to see the grain boundaries. Grains in the microstructure should be fine for as received Al-7046 due to its high strength and grains should be bigger and course for the brazing heat treated Al-7046 due to its less strength compare to as received also due to long time of cooling.

On the other hand, for solution heat treated grains should be finer than brazing heat treated and courser than as received one due to strength recovery.

As shown in the microstructure images, some small particles were seen on the as received samples due to its own structure being alloy with different elements. Also, precipitation hardening phenomenon was occurred for brazing heat treated and solution heat treated Al-7046 alloys.

However, they are many and smaller in brazed heat treated samples comparing to solution heat treated. They are less and bigger in solution heat treated samples. To understand the elements of the particles in these structures, they were analyzed in EDS.

## 4.1.2 Scanning Electron Microscopy (SEM)

The samples of the 1<sup>st</sup> batch of Al-7046 were analyzed with bigger magnifications by SEM.

- **As Received Al-7046**

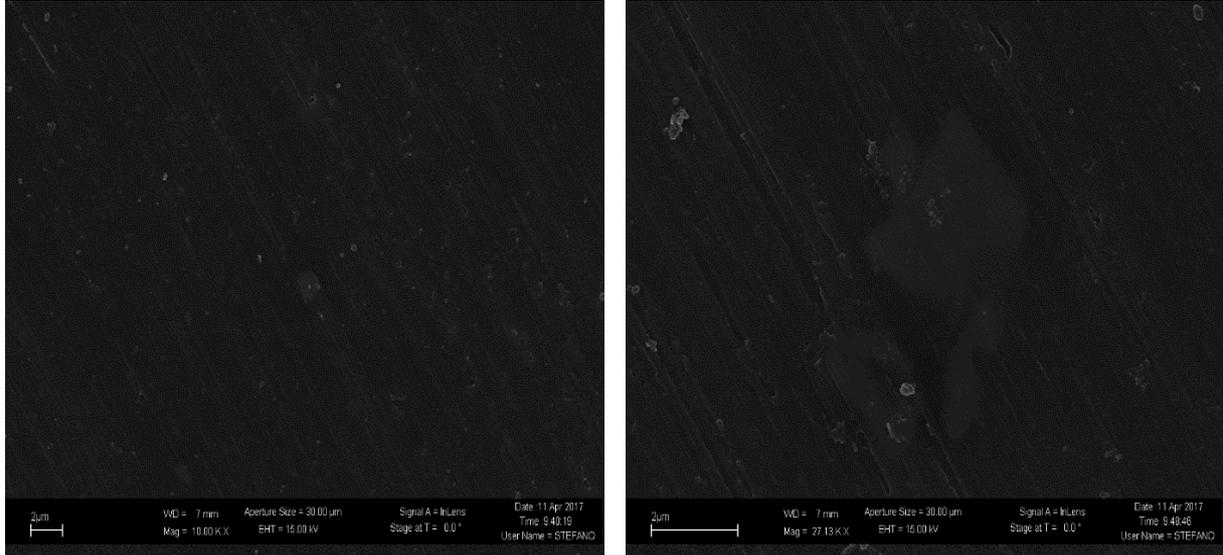


Figure 69: Microstructures of as received Al-7046 magnitude 10KX (on the left) and 27.13KX (on the right), 2µm

- **Brazed Heat Treated Al-7046**

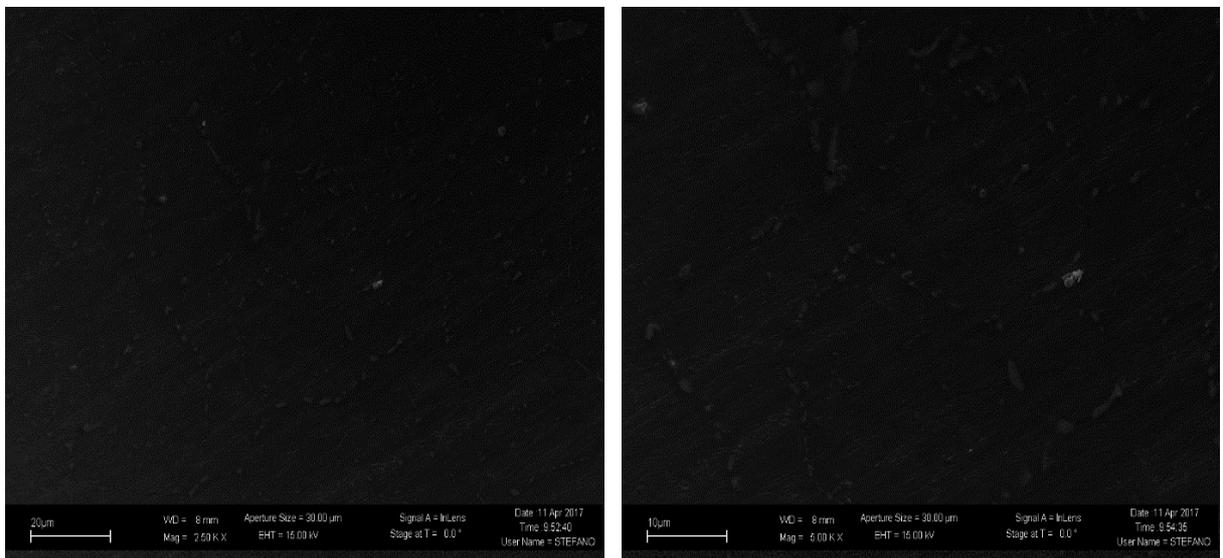


Figure 70: Microstructures of brazed heat treated Al-7046 magnitude 2.50KX, 20µm (on the left) and 5KX, 10µm (on the right)

- **Solution Heat Treated Al-7046**

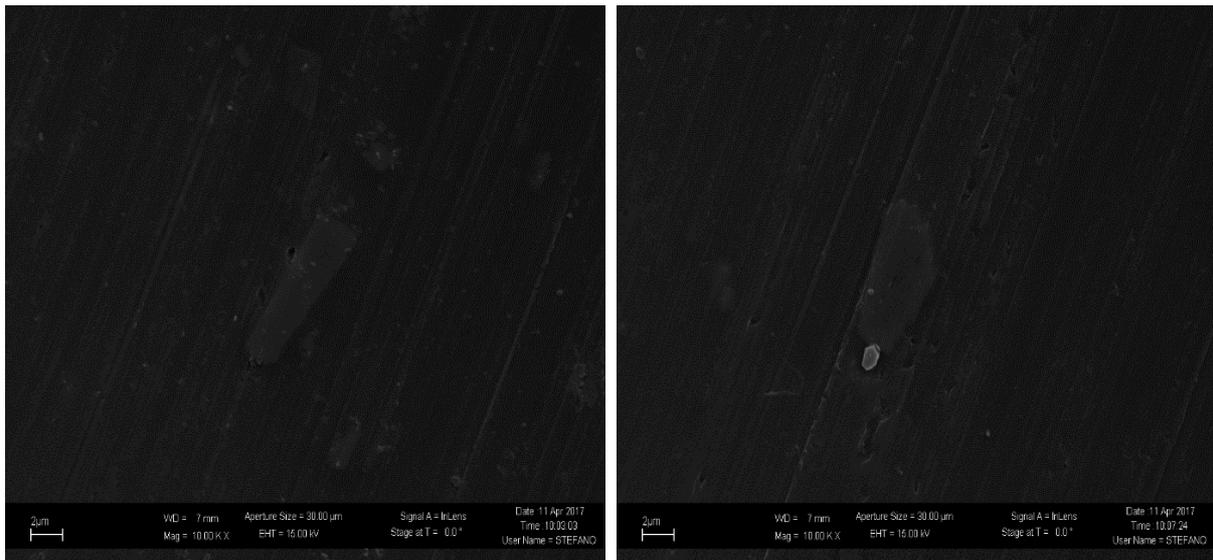


Figure 71: Microstructure of solution heat treated Al-7046 magnitude 10KX, 2µm for both images

In the SEM images of as received Al-7046 alloys, it is observed some small particles due to its own structure being alloy with different elements. However, grain boundaries, needle like points create grain boundaries, are shown in the SEM images of brazing heat treated Al-7046 alloys.

These grains are coarse and big, it is shown better way with 5KX magnitude. These coarse grain boundaries decrease the strength of the material. Unlikely, it is not able to see the grain boundaries in solution heat treated sample. It is observed some dirt and precipitated particles.

### 4.1.3 Energy Dispersive Spectrometry (EDS)

As following to the SEM images, EDS analyzes, for exact position or area on the surface, were conducted for these 3 groups of Al.

- **As Received Al-7046**

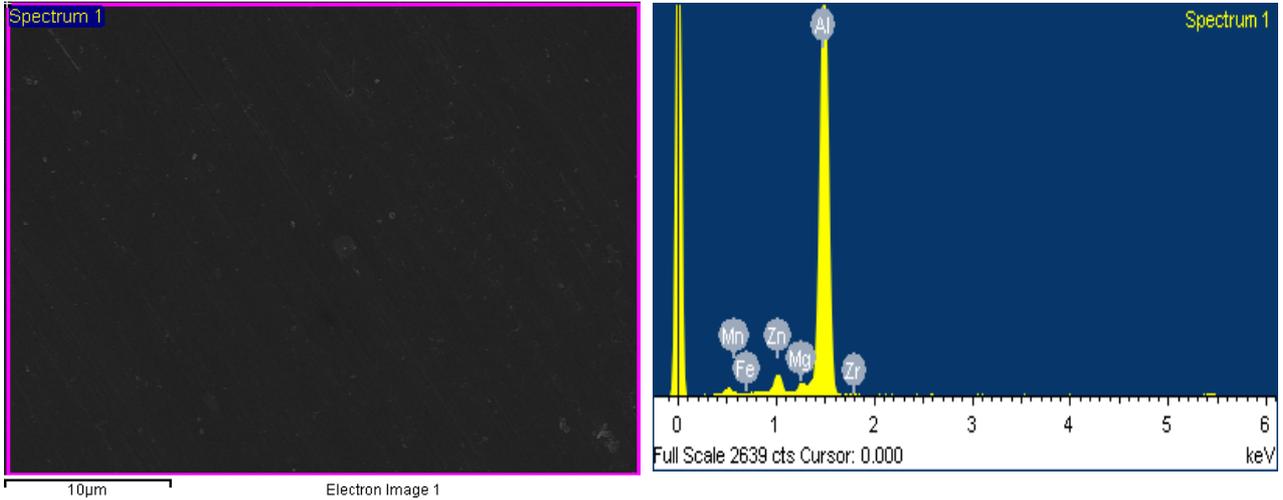


Figure 72: Electron image and EDS spectrum of As Received Al-7046

Table 10: Chemical structure of electron image

Element	Weight%	Atomic%
Mg K	1.53	1.78
Al K	90.16	94.53
Mn K	0.80	0.41
Fe K	0.44	0.22
Zn L	7.07	3.06
Zr L	0.00	0.00
<b>Totals</b>	100.00	

The area of the electron image which obtained from SEM was selected for chemical analyze and the result is reported in the table 10. The main alloying element is Al and Zn is the second following main alloying element.

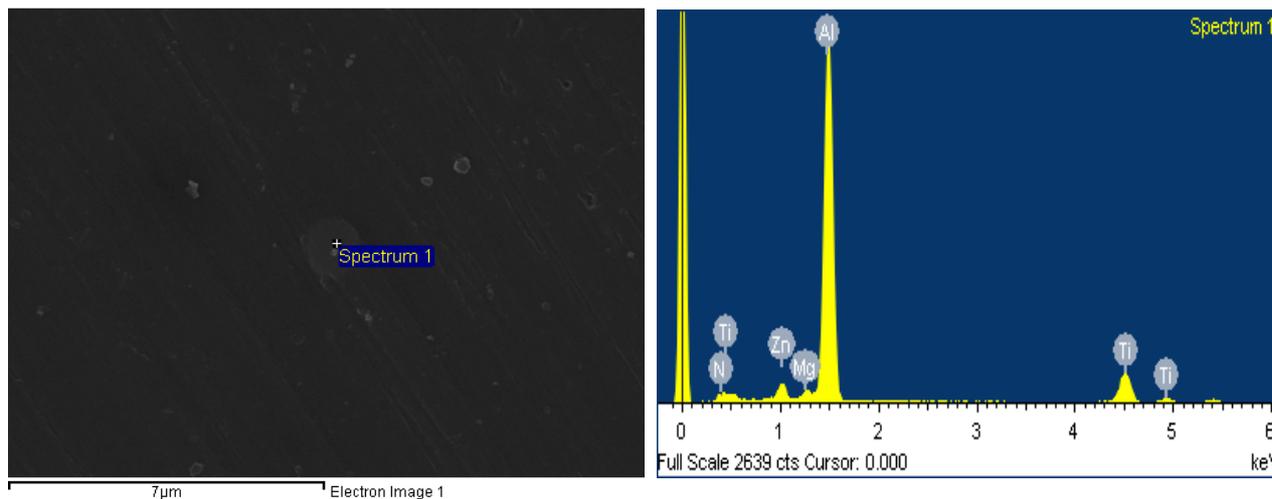


Figure 73: Electron image and EDS spectrum of As Received Al-7046

Table 11: Chemical structure of electron image

Element	Weight%	Atomic%
N K	4.86	10.22
Mg K	1.10	1.33
Al K	66.43	72.51
Ti K	21.27	13.08
Zn L	6.33	2.85
<b>Totals</b>	100.00	

Selected position, spectrum 1, is shown in the figure 73 and chemical analyze of this position is shown in the table 11. There is high amount of Ti in the as received Al-7046 alloy and it might be due to precipitation in the main alloy.

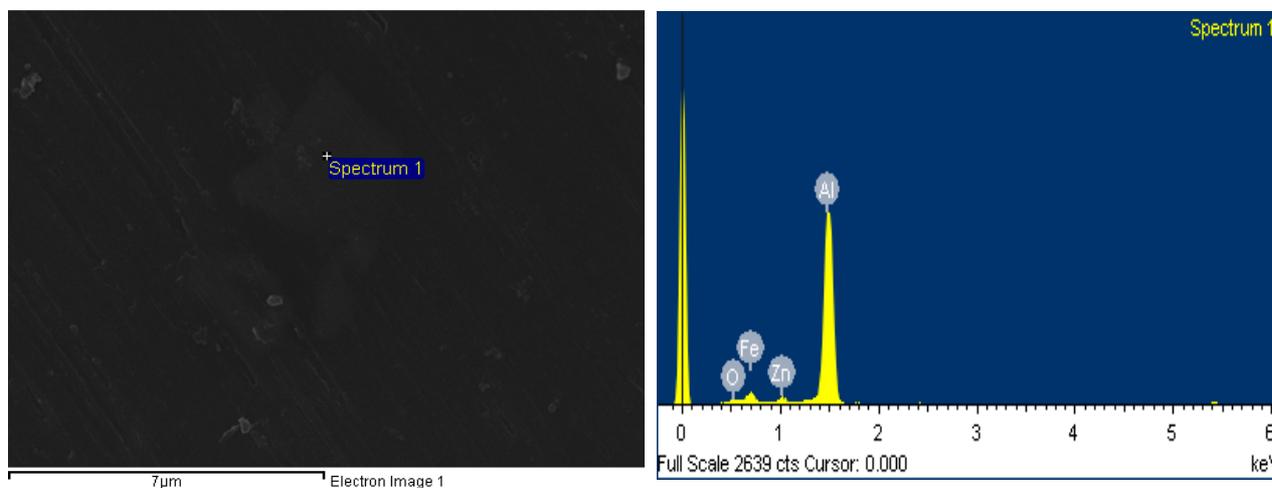


Figure 74: Electron image and EDS spectrum of As Received Al-7046

Table 12: Chemical structure of electron image

Element	Weight%	Atomic%
O K	4.16	7.74
Al K	72.81	80.34
Fe K	18.41	9.82
Zn L	4.62	2.10
<b>Totals</b>	100.00	

Another selected position, spectrum 1, is shown in the figure 74 and chemical analyze of this position is shown in the table 12. There is high amount of Fe in the as received Al-7046 alloy and it might be again due to precipitation in the main alloy.

- **Brazed Heat Treated Al-7046**

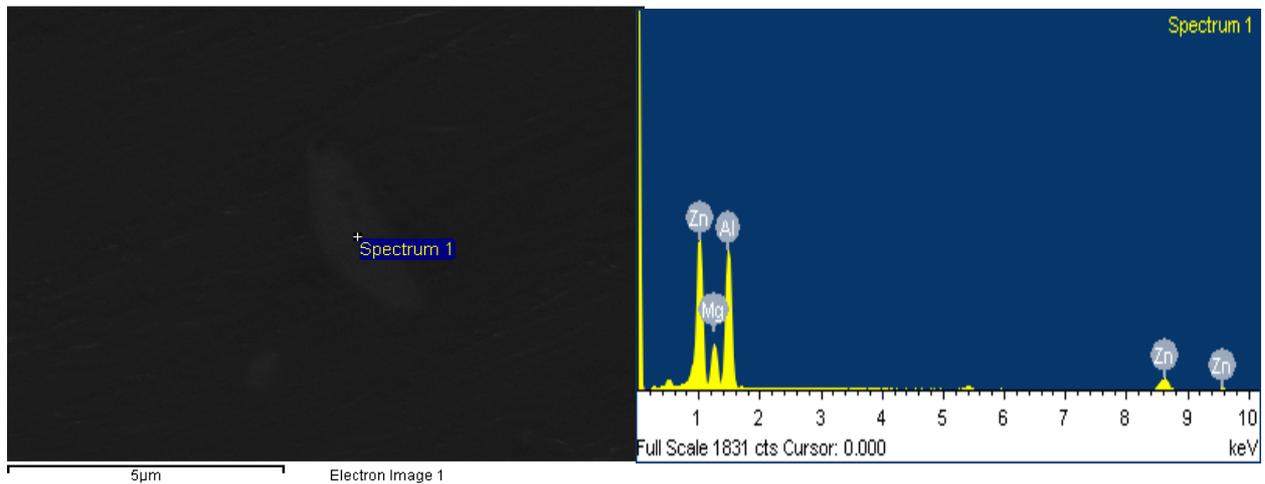


Figure 75: Electron image and EDS spectrum of brazed heat treated Al-7046

Table 13: Chemical structure of electron image

Element	Weight%	Atomic%
Mg K	14.29	21.23
Al K	39.94	53.48
Zn L	45.77	25.29
<b>Totals</b>	100.00	

- **Solution Heat Treated Al-7046**

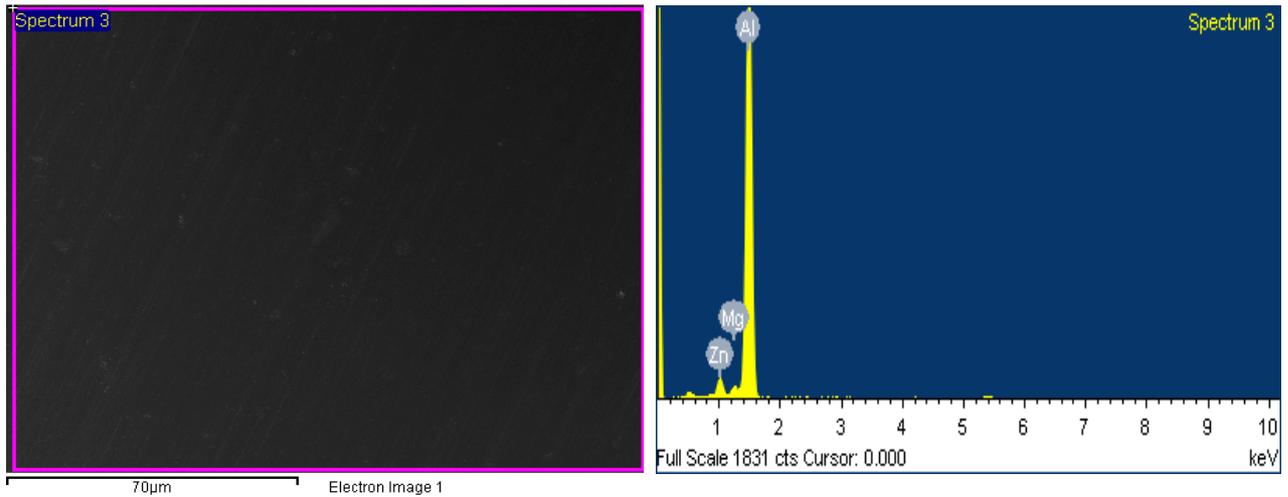


Figure 76: Electron image and EDS spectrum of solution heat treated Al-7046

Table 14: Chemical structure of electron image

Element	Weight%	Atomic%
Mg K	1.72	1.98
Al K	91.54	95.13
Zn L	6.75	2.89
<b>Totals</b>	100.00	

The area was selected, spectrum 3 is shown in the figure 76, for chemical structure analyze. As shown in the table 14, the amount of Al was more appeared and the amount of Zn and Mg were less appeared in the alloy structure after solution heat treatment.

Figure 77: Electron image and EDS spectrum of solution heat treated Al-7046

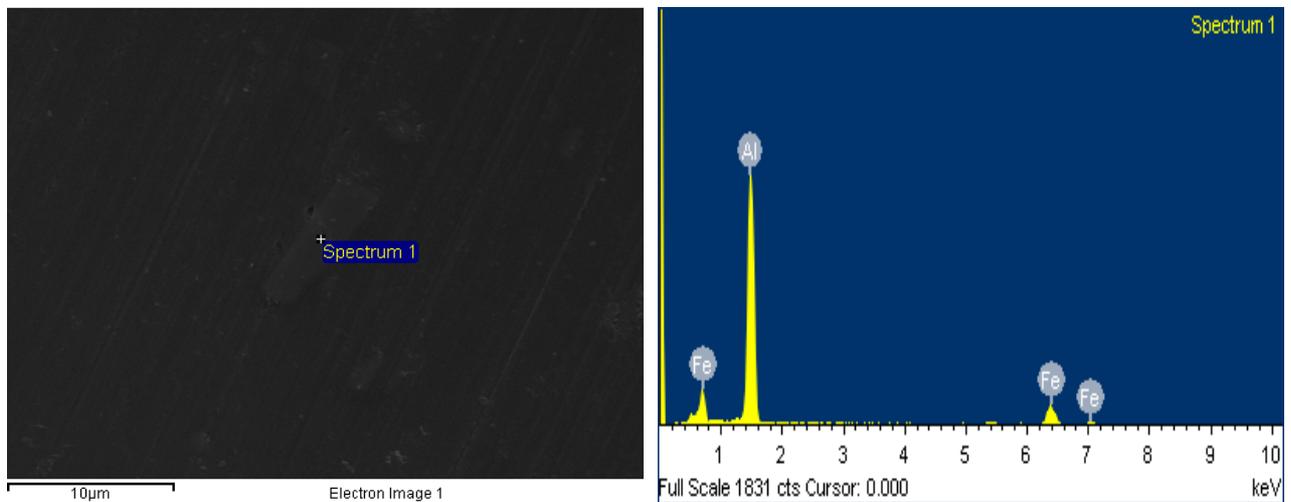


Table 15: Chemical structure of electron image

Element	Weight%	Atomic%
Al K	64.17	78.75
Fe K	35.83	21.25
<b>Totals</b>	100.00	

Selected position was inside the area of the second phase precipitation, spectrum 1 is shown in the figure 77. The chemical structure of this precipitation is shown in the table 15 and high amount of Fe precipitation was shown in solution heat treated Al-7046 alloy.

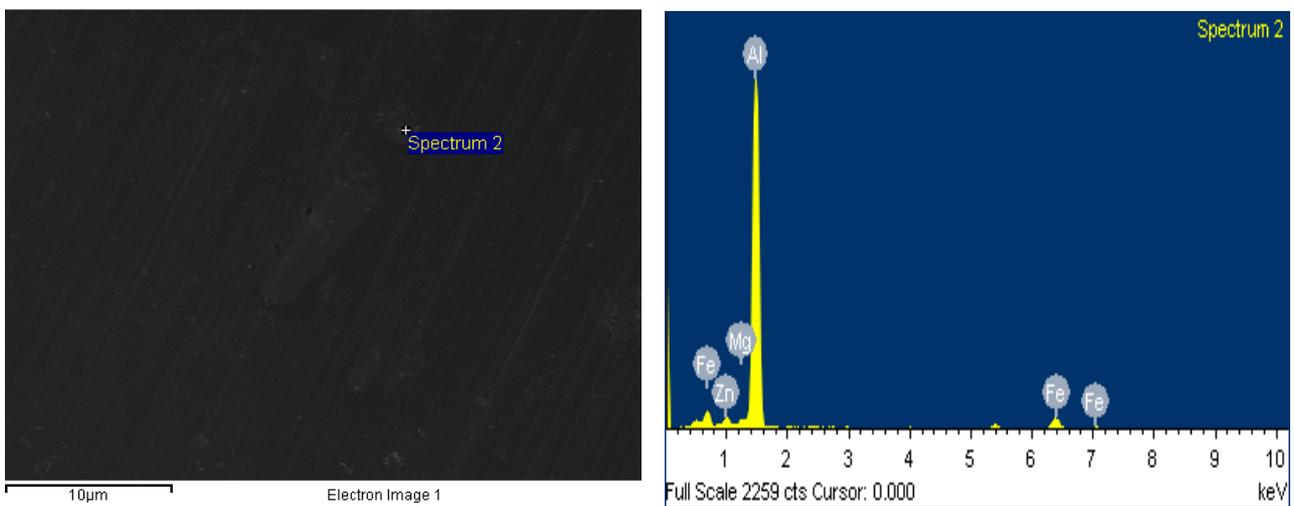


Figure 78: Electron image and EDS spectrum of solution heat treated Al-7046

Table 16: Chemical structure of electron image

Element	Weight%	Atomic%
Mg K	0.97	1.22
Al K	75.23	85.98
Fe K	19.48	10.76
Zn L	4.32	2.04
<b>Totals</b>	100.00	

Another selected position was inside another phase precipitation, spectrum 2 is shown in the figure 78, which mixed phase of the base structure and second phase. The mixture phase of base structure and second phase is shown in the table 16. The amount of Fe became less active while there was an increase activity of Zn amount. It may said that there are different phases in the solution heat treated Al-7046 alloy such as base structure, second phase precipitation and mixed phase between base and second phase.

### 4.1.4 Micro Hardness Test

The Vickers micro hardness test were performed for as received (A, AR), brazed heat treated (B) and solution heat treated (C, SH) Al-7046 alloys, shown in the figure 79.

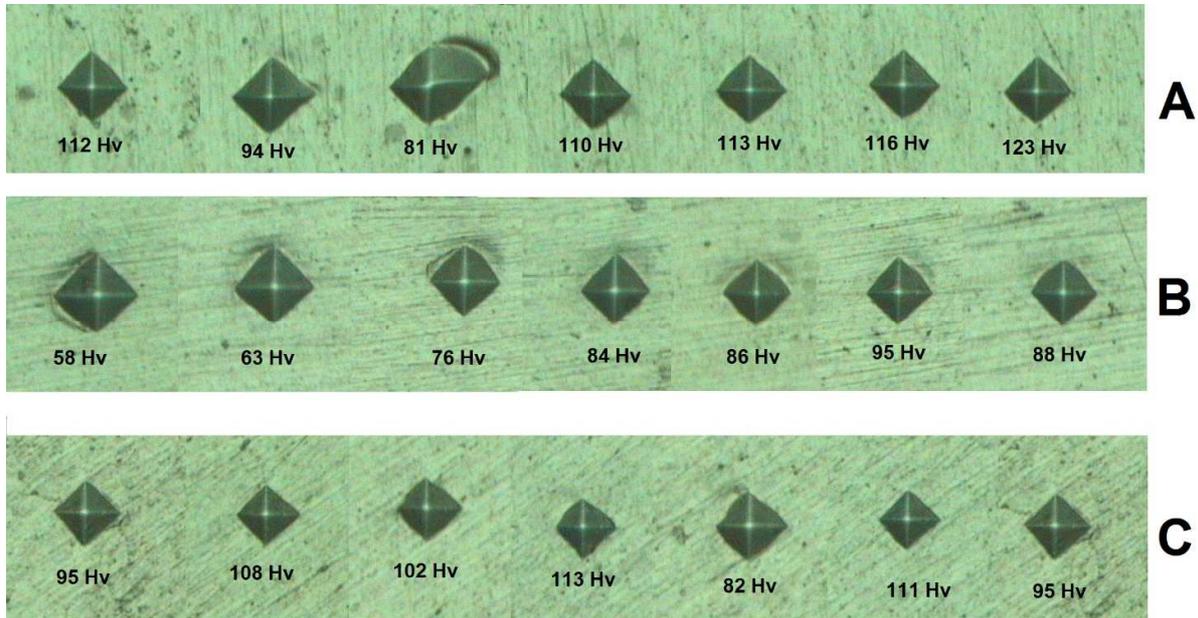


Figure 79: Micro hardness test results for 1<sup>st</sup> batch Al-7046 alloys

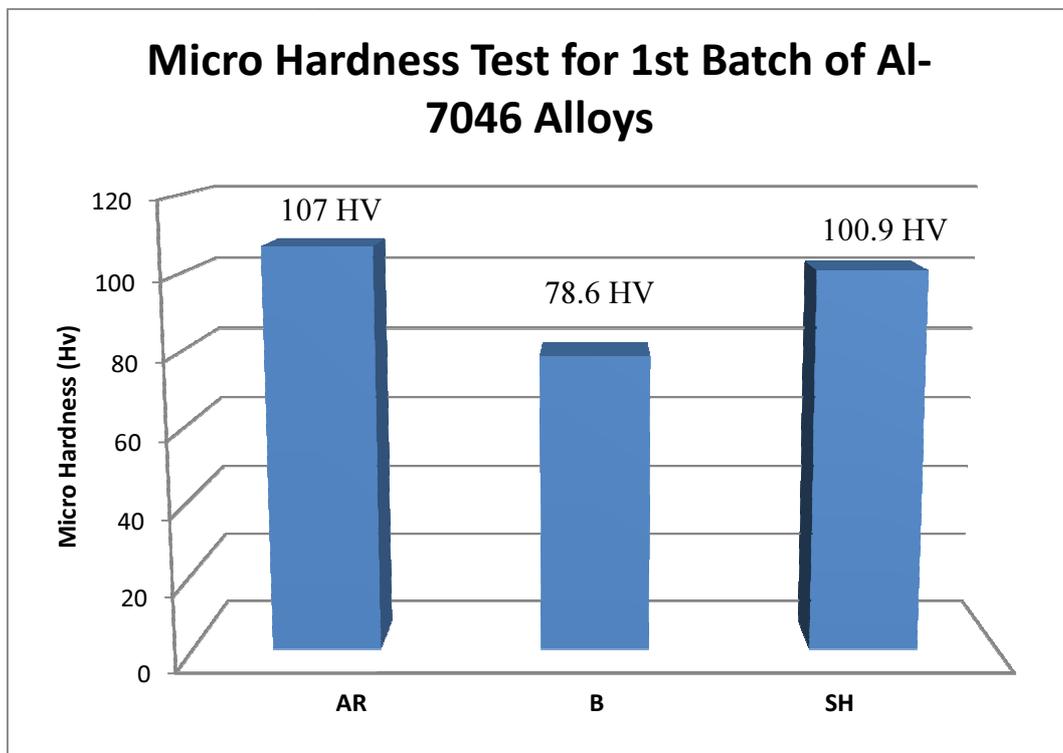


Figure 80: Micro hardness test results for 1<sup>st</sup> batch Al-7046 alloys and their average hardness values

As reported on the average hardness values in the figure 80, the maximum hardness was obtain for as received Al-7046 alloy then solution heat treated and the minimum hardness for brazed heat treated Al-7046 alloy. It was expected to have decrease of strength and hardness after brazing heat treatment due to long time of cooling and microstructures that grain boundaries became coarser and bigger.

As following to the solution heat treatment after brazing heat treatment, good amount of hardness was recovered. Thus, solution heat treatment was beneficial for recovering the micro hardness of the Al-7046 alloys after brazing heat treatment.

### 4.1.5 3-Point Bending Flexural Test

3 point bending flexural test were performed for the as received (AR), brazed heat treated (B) and solution heat treated (SH) specimens as following;

- Specimen Size

Table 17: Specimen features of 1<sup>st</sup> batch of Al- 7046 alloys

<b>Specimen Name</b>	<b>Length (mm)</b>	<b>Width (mm)</b>	<b>Thickness (mm)</b>
AR1	60	20	1.28
AR2	59.96	19.92	1.28
AR3	59.94	19.95	1.28
B1	59.97	19.99	1.28
B2	60	19.91	1.28
B3	60	19.95	1.28
SH1	60	19.97	1.28
SH2	60	19.95	1.28
SH3	59.98	19.98	1.28

- Span Length = 47 mm
- Loading Speed = 4mm/min
- Number of Specimens = 9 (three specimens for each category).

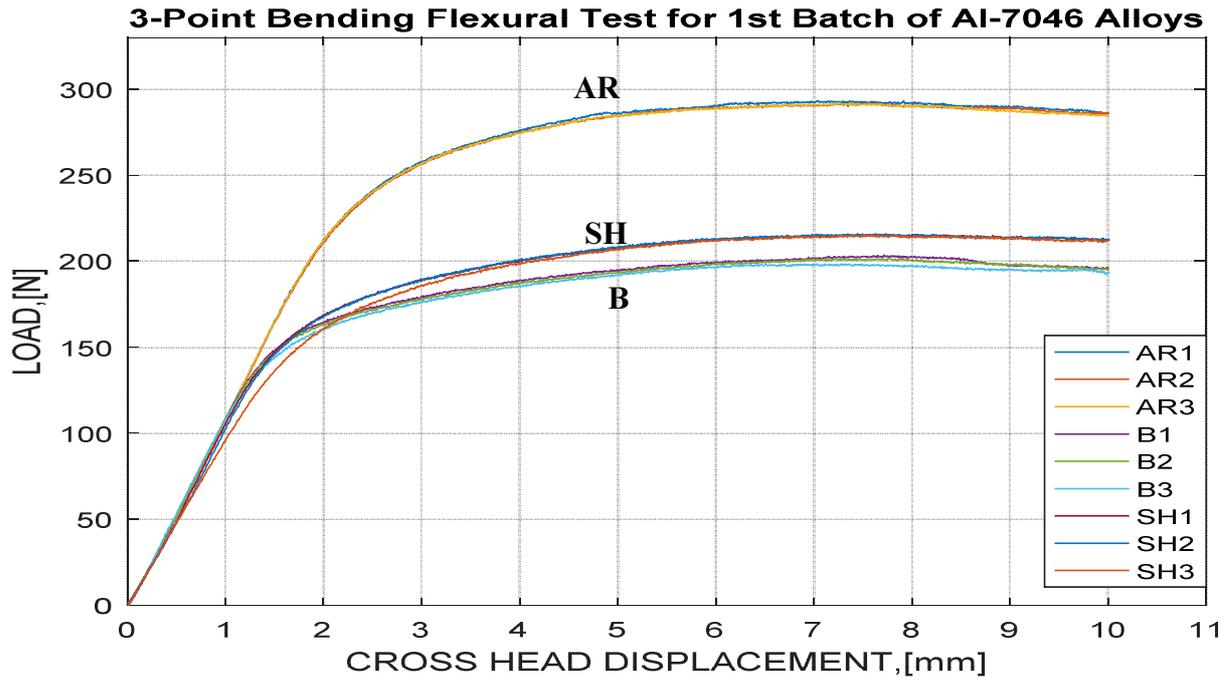


Figure 81: 3 point bending flexural test results for 1<sup>st</sup> batch of Al-7046 alloys

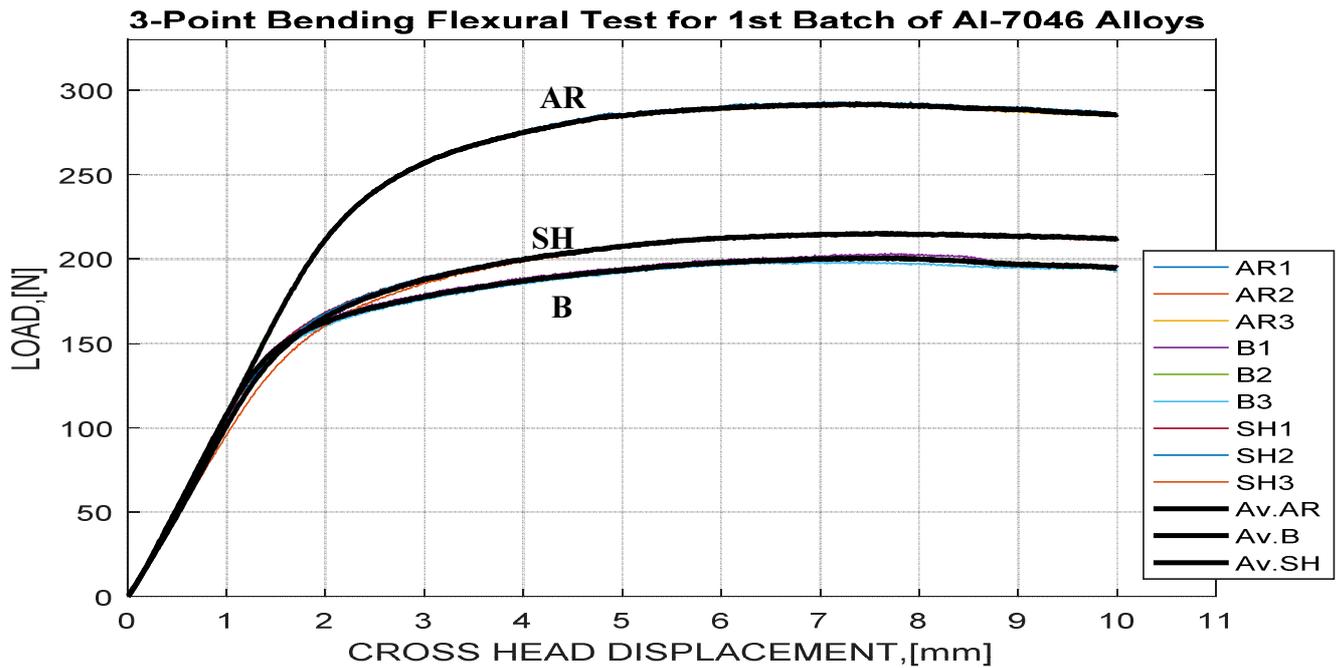


Figure 82: 3 point bending flexural test average values for 1<sup>st</sup> batch of Al-7046 alloys

As reported in the figure 81 that the maximum strength was obtained for as received Al-7046 alloys, it was around 280 N. Referring to the literature that there should be decrease of strength after brazing heat treatment, there was decrease of strength after brazing heat treatment around 28% due to long cooling time on the treatment and their microstructure became coarser after this treatment. Solution heat treatment was performed as recovery the strength and it is observed 10% increase of brazing heat treated strength.

Also, average curves of these 3 samples from each groups are reported in the figure 82, it is observed the benefit of doing solution heat treatment to the samples after brazing heat treatment. It was also observed the positive effects of solution heat treatment in terms of strength and mechanical properties.

For each sample, the other 3 point bending flexural test results such as elastic modulus and samples physical properties such as dimensions and weight are shown below.

Table 18: Elastic modulus and physical properties of 1<sup>st</sup> batch Al-7046 alloy

Specimen	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	E-Mod (GPa)
AR1	60	20	1.28	4.2191	71.486
AR2	59.96	19.92	1.28	4.2044	70.971
AR3	59.94	19.95	1.28	4.2091	71.389
B1	59.97	19.99	1.28	4.2604	71.670
B2	60	19.91	1.28	4.2034	70.480
B3	60	19.95	1.28	4.2209	71.022
SH1	60	19.97	1.28	4.243	70.705
SH2	60	19.95	1.28	4.242	65.163
SH3	59.98	19.98	1.28	4.2423	61.143

The average elastic modulus of as received and brazed heat treated Al-7046 alloys were almost the same, reported in the figure 83. There was 7.6% decrease of elastic modulus on solution heat treated Al-7046 samples. It may said that as received and brazed heat treated Al-7046 alloys have more resistance to being deformed elastically than solution heat treated samples when a stress is applied to it.

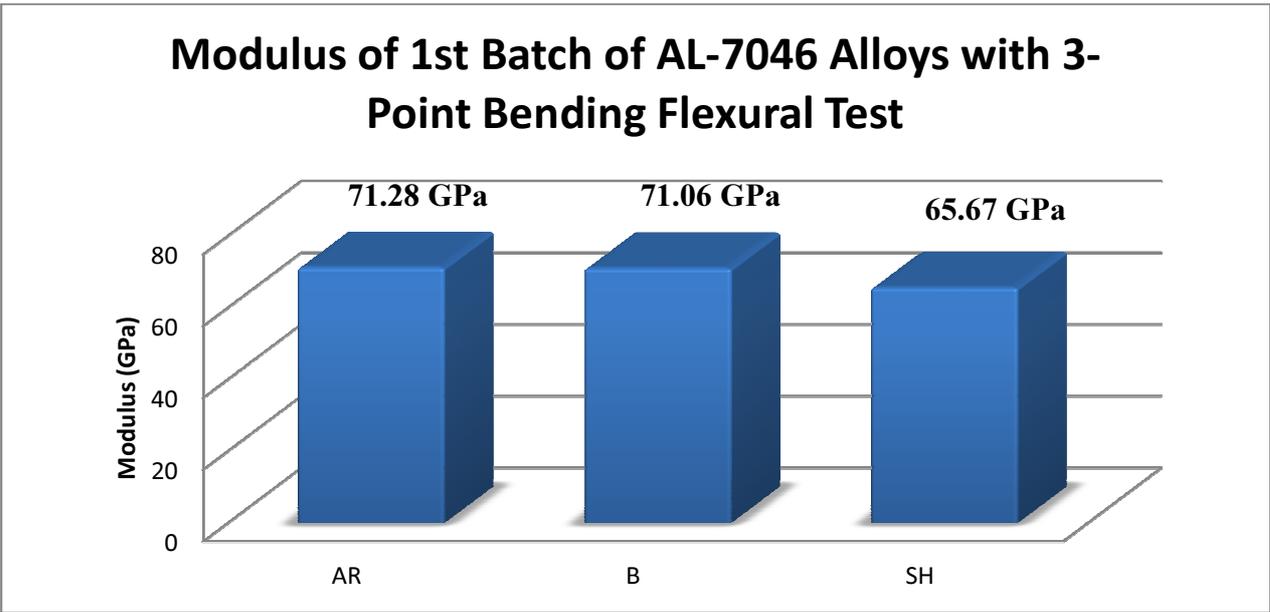


Figure 83: Modulus of 1<sup>st</sup> batch Al-7046 alloys with 3 point bending flexural test

## 4.2 AFS Composite Brazed with Pure Zn

AFS composites in this title consist of two groups as braze joining with pure Zn and following to braze joining with pure Zn, solution heat treated AFS composites.

### 4.2.1 3-Point Bending Test

3 point bending flexural test were performed for the AFS composites which brazed with pure Zn. B represents only being brazed without solution heat treatment and SH represents being brazed then solution heat treated following to braze joining treatment. The numbers on the each group after letter represent the identity of the specimens as following;

- Specimen Size

Table 19: Specimen features of AFS composite with pure Zn

Specimen	Length (mm)	Width (mm)	Thickness (mm)
B1	62.7	21.38	11.4
B7	62.3	23	11.24
B4	63.4	22.8	11.45
SH2	64.5	24.1	11.4
SH6	62.2	23.7	11.5
SH5	63	24.2	11.6

- Span Length = 47 mm
- Loading Speed = 4mm/min
- Total 3 samples brazing heat treated, 3 samples brazing heat treated after solution heat treated

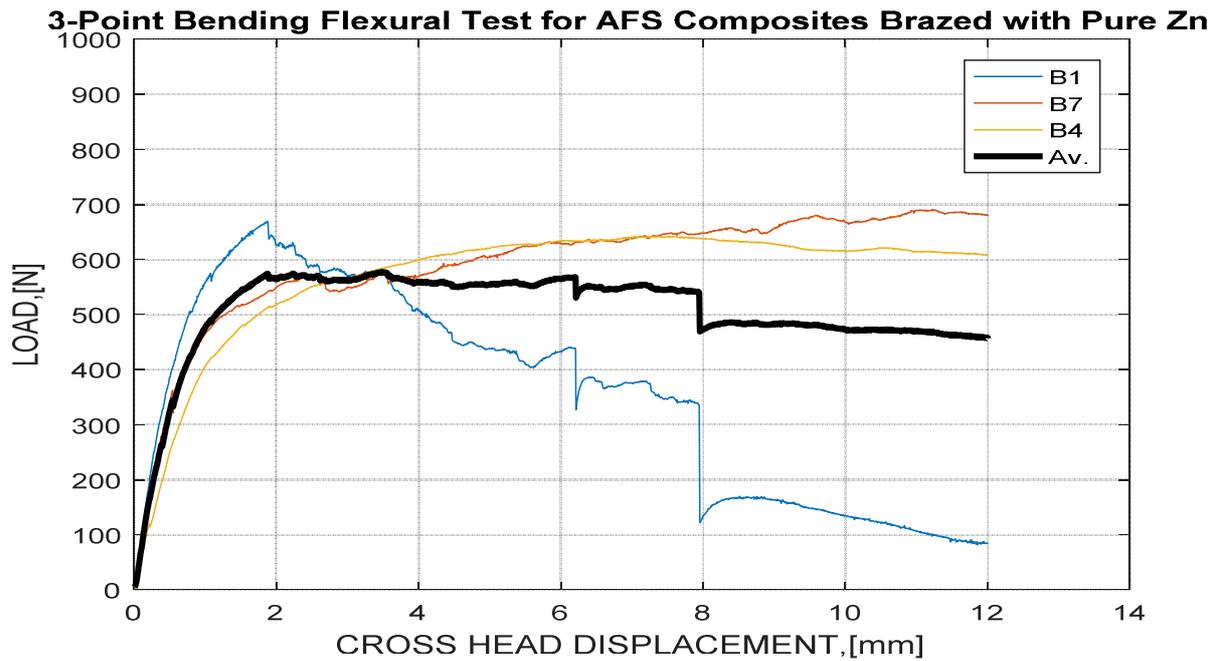


Figure 84: 3 point bending flexural test results for AFS composites brazed with pure Zn

All 3 brazed heat treated samples are reported in the figure 84 and load drops were observed on the samples due to little cracks in Al-foam or on the Al plate or separation from the joining seam. Also, the maximum load was obtained around 680N for the samples. The black curve represents the trend of load on cross head displacement average of these 3 brazed heat treated curves.

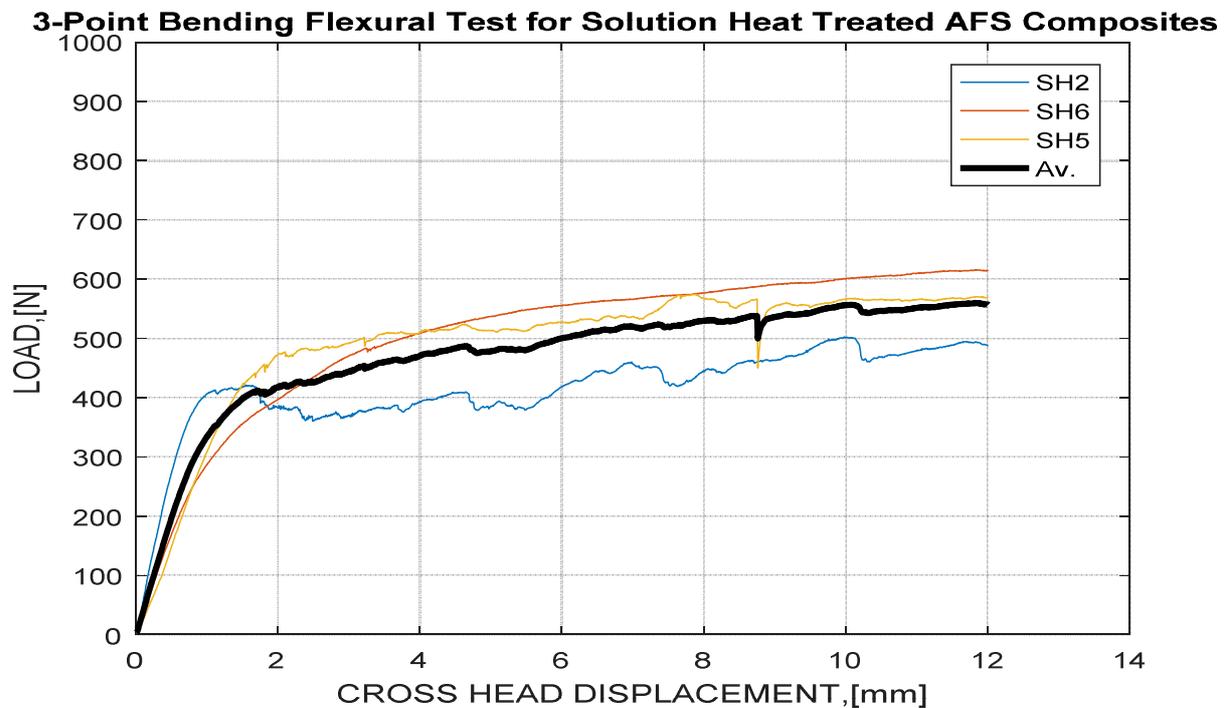


Figure 85: 3 point bending flexural test results for solution heat treated AFS composites brazed with pure Zn

All 3 solution heat treated samples are reported in the figure 85 and big load drop was observed on the sample number SH5 when the cross head displacement came to around 8.7mm. At that point, there was a crack on the sample. Also, other little load drops were seen on the all samples due to separation of joining seams or Al foam behaviour. The maximum load around 600N was obtained during the test. Black curve shows the average of these 3 solution heat treated curves.

Unlikely to the 1<sup>st</sup> batch that it was seen the positive effects of solution heat treatment to the brazed samples in terms of strength, it did not work good for AFS composites in order to increase the strength after brazing joining treatment. When the maximum load was around 680N for brazed joint for AFS composite, whereas 600N maximum load was obtained for solution heat treated sample. Thus, the positive effect that was seen on 1<sup>st</sup> batch Al-7046 alloys was not obtained on the AFS composites when solution heat treatment was applied.

When two average curves of brazed and solution heat treated AFS composites were compared, reported in the figure 86, when the maximum load of brazed AFS composite was 590N meanwhile the maximum load of solution heat treated AFS composite was remained less than 590N. It was also witnessed that there is no beneficial effect of conducting solution heat treatment on the AFS composites in terms of strength due to foam structure of the AFS composites.

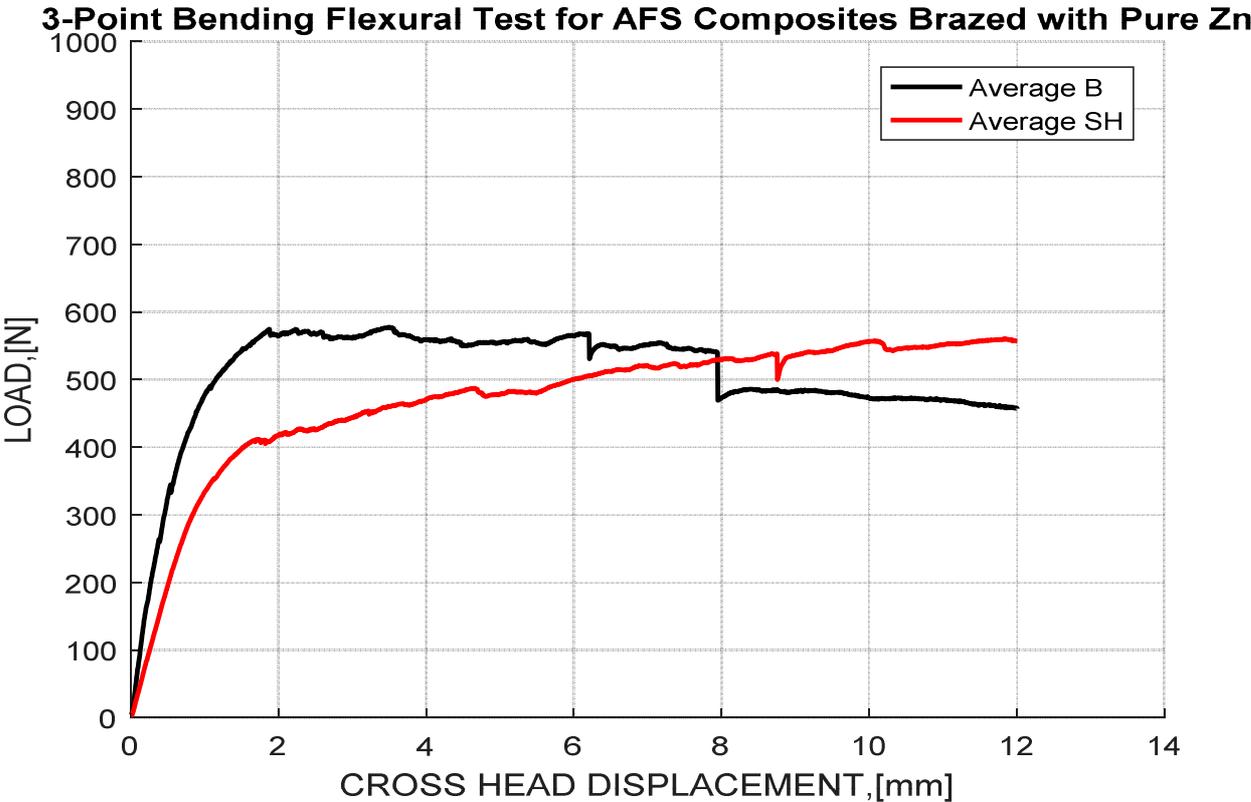


Figure 86: Average results of brazed and solution heat treated AFS composite

## Elastic Modulus

The results of elastic modulus were obtained from 3 point bending flexural test and also physical properties such as dimension and weight for each AFS composites are reported below;

Table 20: Elastic modulus and physical properties of AFS composite brazed with pure Zn

Specimen	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	E-Mod (MPa)
B1	62.7	21.38	11.4	14.5	779
B7	62.3	23	11.24	14.62	755
B4	63.4	22.8	11.45	15.35	423
SH2	64.5	24.1	11.4	15.04	463
SH6	62.2	23.7	11.5	15.96	252
SH5	63	24.2	11.6	16.96	241

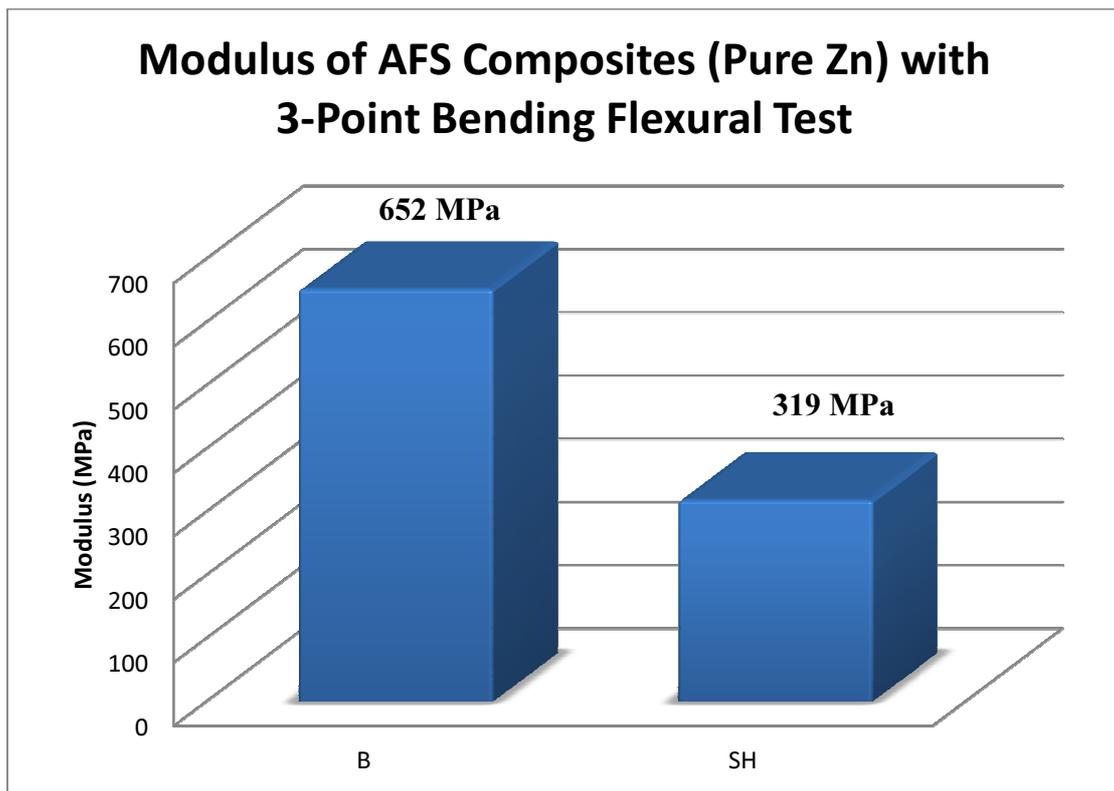


Figure 87: Modulus of AFS composites brazed pure Zn with 3 point bending flexural test

The average modulus values of brazed and solution heat treated AFS composites were reported in the figure 87 and only brazed AFS composites have got almost double value of solution heat treated one and they have more resistance to being deformed elastically.

## 4.2.2 Micro Hardness Test

The Vickers micro hardness test were performed for brazed AFS composite with pure Zn and solution heat treated AFS composite.

- **Brazed AFS composite**

There were regions of diffusion zone, joint between plate and foam, plate and foam side.

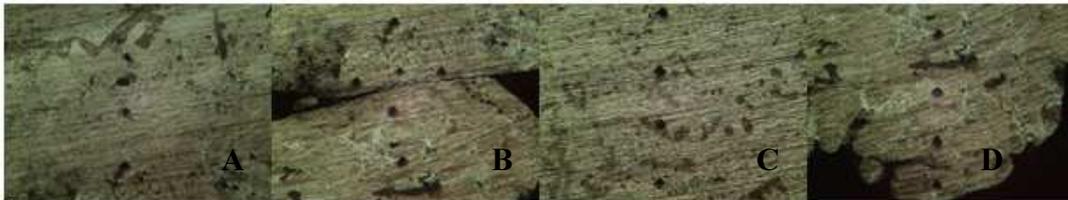


Figure 88: Diffusion zone (A), joint (B), plate (C), foam (D)

- Al-7046 plate edge of the joining seam of the AFS composite

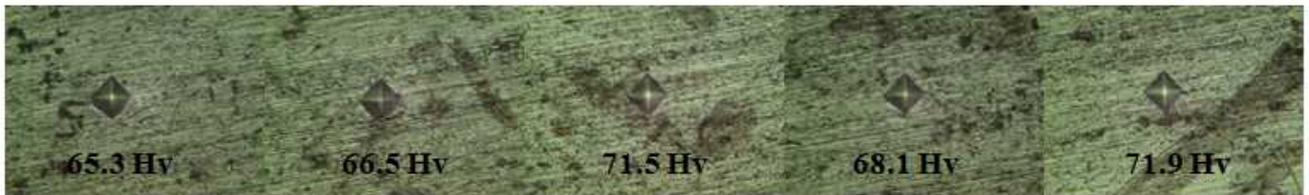


Figure 89: Micro Hardness of Al-plate edge of the joining seam of AFS composite

- Diffusion zone in the joining seam of the AFS composite



Figure 90: Hardness of diffusion zone in the joining seam of AFS composite

- Al-Foam on the joining seam of AFS composite



Figure 91: Micro Hardness of Al-Foam on the joining seam of AFS composite

- Joint seam of AFS composite



Figure 92: Micro Hardness of joint seam of AFS composite

Again it was observed that the hardness was increased in the diffusion zone comparing with the plate region in the figure 93.

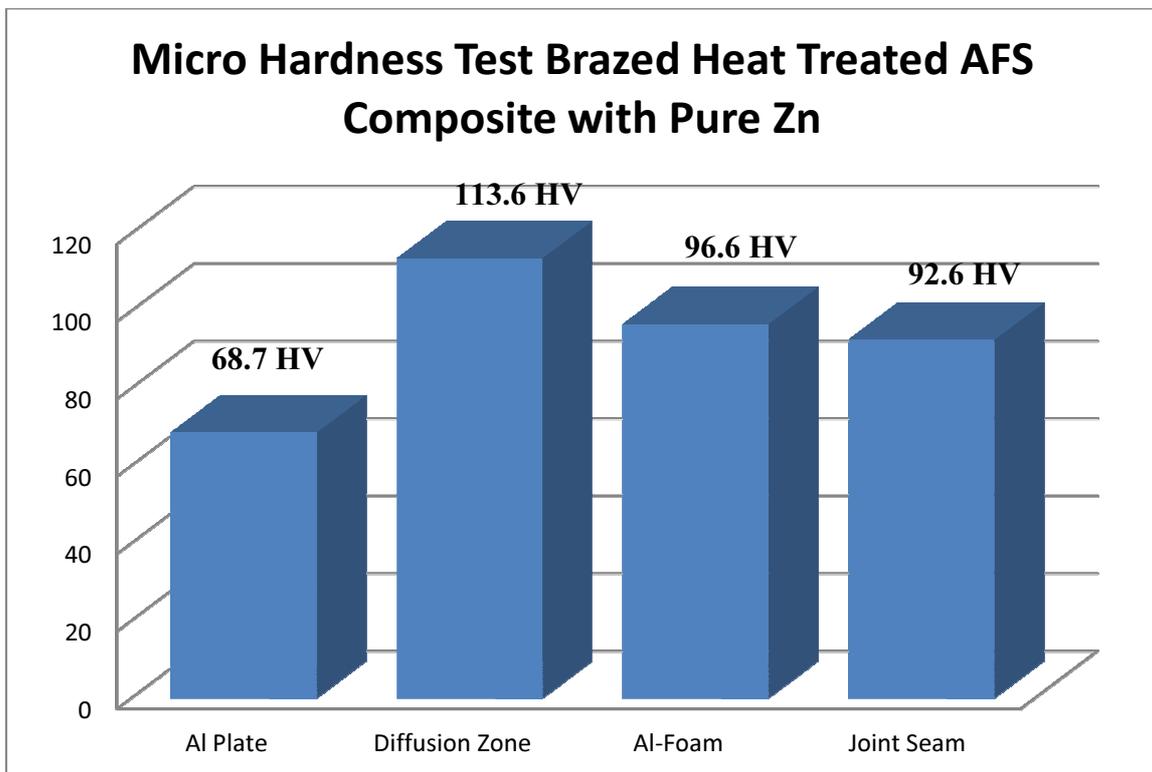


Figure 93: Micro hardness average results of brazed AFS composite with pure Zn

- **Solution Heat Treated AFS composite**

There were regions of diffusion zone, joint between plate and foam, plate and foam side.

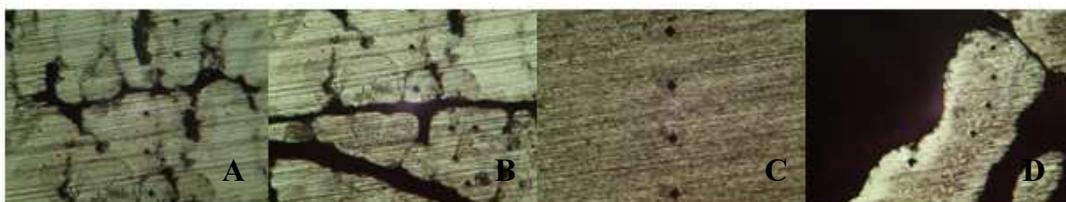


Figure 94: Diffusion zone (A), joint (B), plate (C), foam (D)

- Al-7046 plate edge of the joining seam of AFS composite



Figure 95: Micro Hardness of Al-plate edge of the joining seam of AFS

- Diffusion zone in the joining seam of the AFS composite



Figure 96: Hardness of diffusion zone in the joining seam of AFS composite

- Al-Foam on the joining seam of AFS composite



Figure 97: Micro Hardness of Al-Foam on the joining seam of AFS composite

- Joint seam of AFS composite

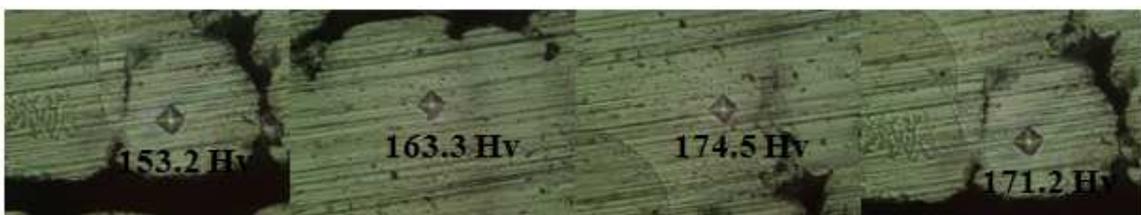


Figure 98: Micro Hardness of joint seam of AFS composite

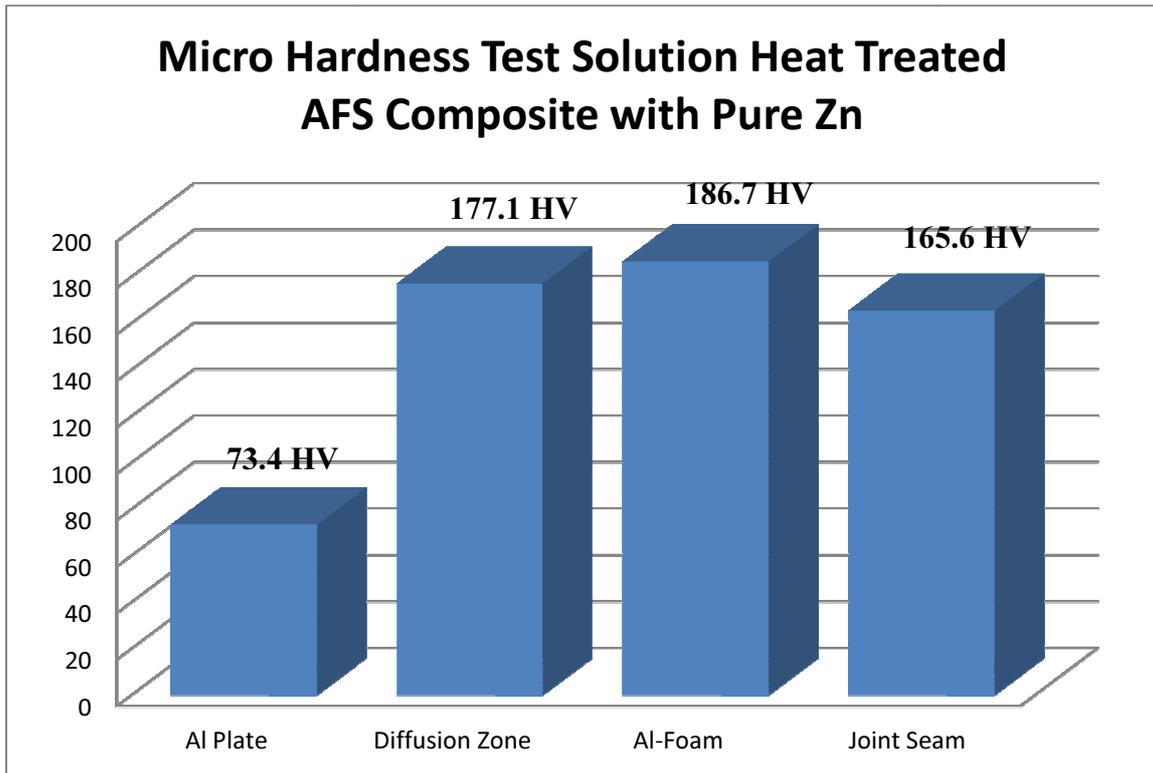


Figure 99: Micro hardness average results of solution heat treated AFS composite with pure Zn

As reported in the figure 99, micro hardness average value was increased on the diffusion zone and on the foam side again. Comparing to the brazed AFS composite, it was observed the increase of hardness from 68.7 to 73.4 HV on Al plate edge of the joining seam, from 113.6 to 177.1 HV on diffusion zone, from 96.6 to 186.7 HV in Al-foam on the joining area, from 92.6 to 165.6 HV on the joint seam when solution heat treatment was performed on the AFS composites. It would be said that the hardness values all of the parts of the composite were increased with solution heat treatment.

### 4.3 AFS Composite Brazed with Zn2%Al

AFS composites in this title consist of two groups as braze joining with Zn2%Al and following to braze joining with Zn2%Al, solution heat treated AFS composites.

#### 4.3.1 3-Point Bending Test

3 point bending flexural test were performed for the AFS composites which brazed with Zn2%Al. B represents only being brazed without solution heat treatment and SH represents being brazed then solution heat treated following to brazing joining treatment. The numbers on the each group after letter represent the identity of the specimens as following;

- Specimen Size

Table 21: Specimen features of AFS composite with pure Zn

Specimen	Length (mm)	Width (mm)	Thickness (mm)
B1	62.5	22.7	11.3
B5	61.7	21.9	11.17
B6	63.6	22.4	10.5
SH2	61.9	23.1	11.1
SH3	61.3	22.9	11
SH4	61.3	23.3	11.5

- Span Length = 47 mm, Loading Speed = 4mm/min.
- Total 3 samples brazing heat treated, 3 samples brazing heat treated after solution heat treated

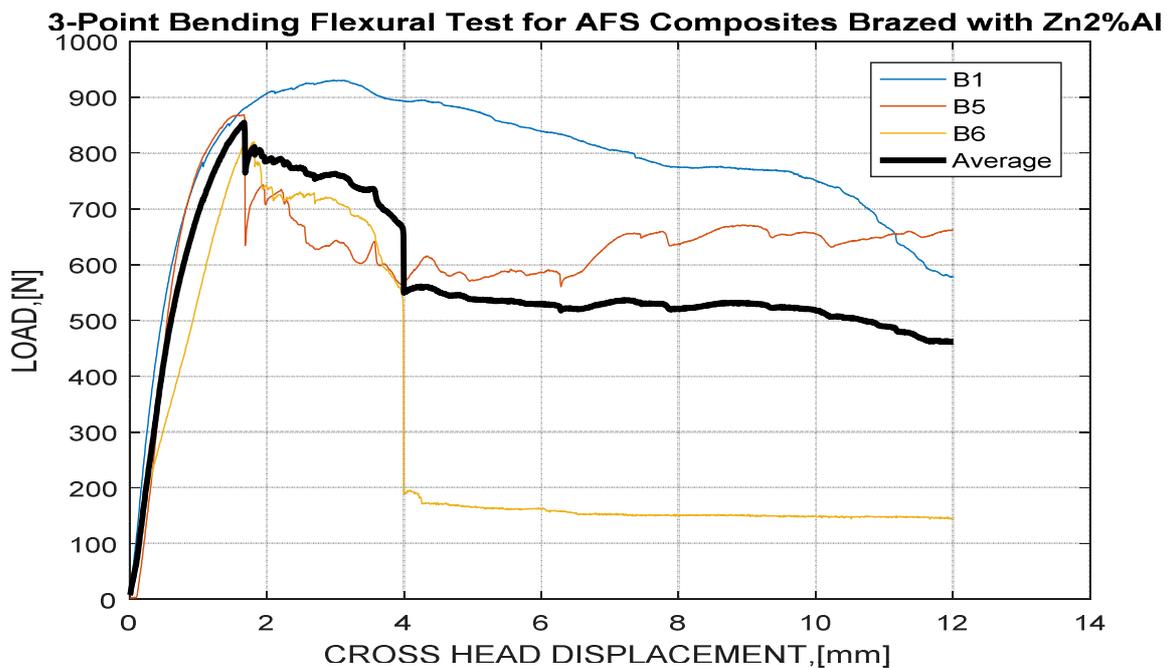


Figure 100: 3 point bending flexural test results for AFS composites brazed with Zn2%Al

All 3 brazed heat treated samples are reported in the figure 100 and load drops were seen on the samples due to little cracks in Al-foam or on the Al plate or separation from the joining seam. Also, the maximum load was obtained around 920N for samples whereas maximum load was 680N for AFS composites brazed with pure Zn. It may said that using Zn2%Al as brazing material is better application rather than using pure Zn in terms of strength. There was 35% increase of the maximum load when Zn2%Al was used as brazing material. The black curve represents the trend of load on cross head displacement average of these 3 brazed heat treated curves.

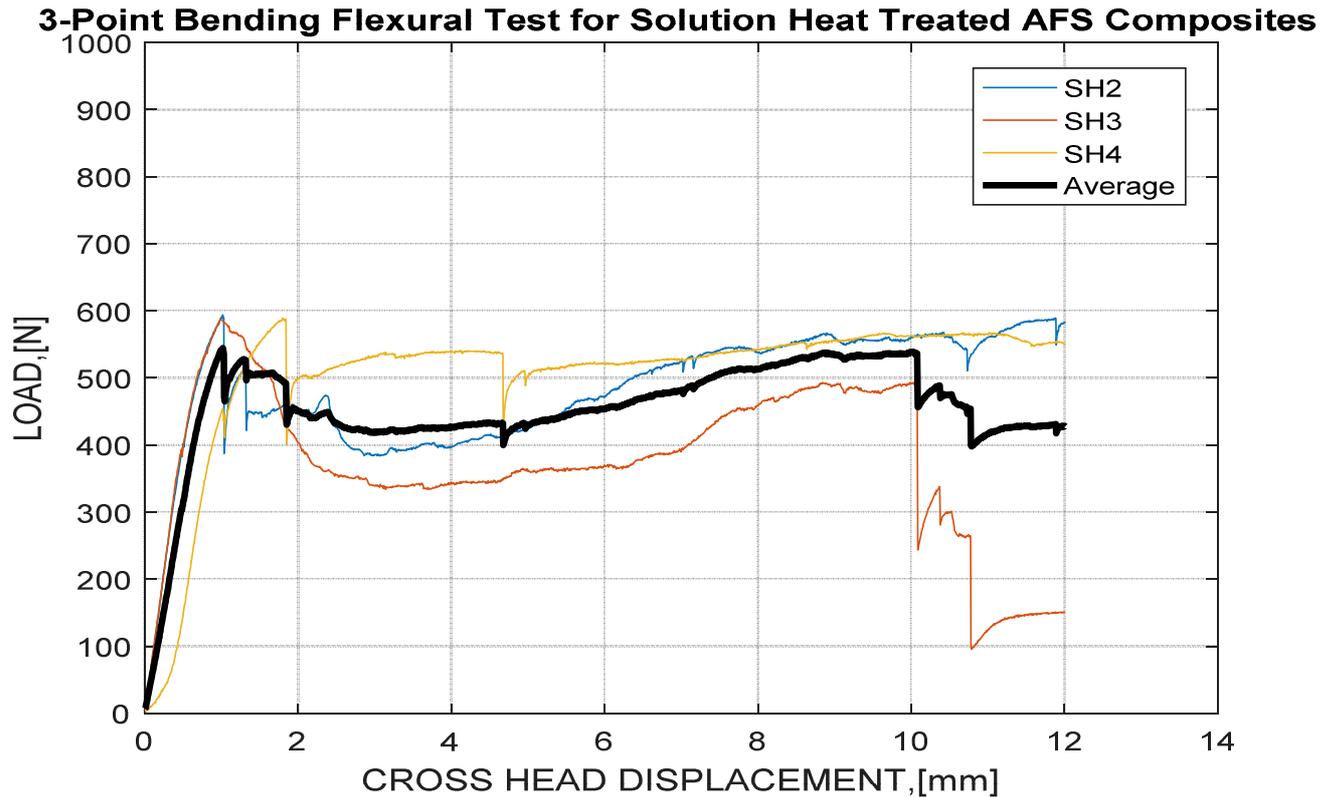


Figure 101: 3 point bending flexural test results for solution heat treated AFS composites brazed with Zn2%Al

All 3 solution heat treated samples are reported in the figure 101. It was witnessed one side separation of the joint seam between Al-foam and Al plate and cracks on the composite during bending test. The maximum load around 600N was obtained for solution heat treated samples during the test. Black curve shows the average of these 3 solution heat treated curves.

Unlikely again to the 1<sup>st</sup> batch that it was seen the positive effects of solution heat treatment to the brazed samples in terms of strength, it did not work good for AFS composites brazed with Zn2%Al in order to increase the strength after brazing joining treatment. When the maximum load was around 920N for brazed joint for AFS composite with Zn2%Al, whereas 600N maximum load was obtained for solution heat treated sample.

Even though solution heat treatment was applied in order to increase the mechanical properties such as strength and maximum load capacity, it was observed decrease of 35% on maximum load after solution heat treatment. Thus, again the positive effect that was witnessed on 1<sup>st</sup> batch Al-7046 alloys was not obtained on the AFS composites brazed with Zn2%Al when solution heat treatment was applied.

When two average curves of brazed and solution heat treated AFS Zn2%Al composites were compared, reported in the figure 102, when the maximum load of brazed AFS composite was 860N meanwhile the maximum load of solution heat treated AFS composite was remained less than 550N. It was also witnessed again that there is no beneficial effect of conducting solution heat treatment on the AFS Zn2%Al composites in terms of strength due to foam structure of the AFS composites.

However, using Zn2%Al as brazing material was better option to apply rather than pure Zn as brazing material because when Zn2%Al was used, the maximum average load capacity was 860N. Meanwhile maximum average load capacity was 590N when pure Zn was used. It can be said that there was 45% increase of maximum load capacity when AFS composites were brazed with Zn2%Al. There was no good effect of doing solution heat treatment in terms of maximum load and strength for both AFS composites groups.

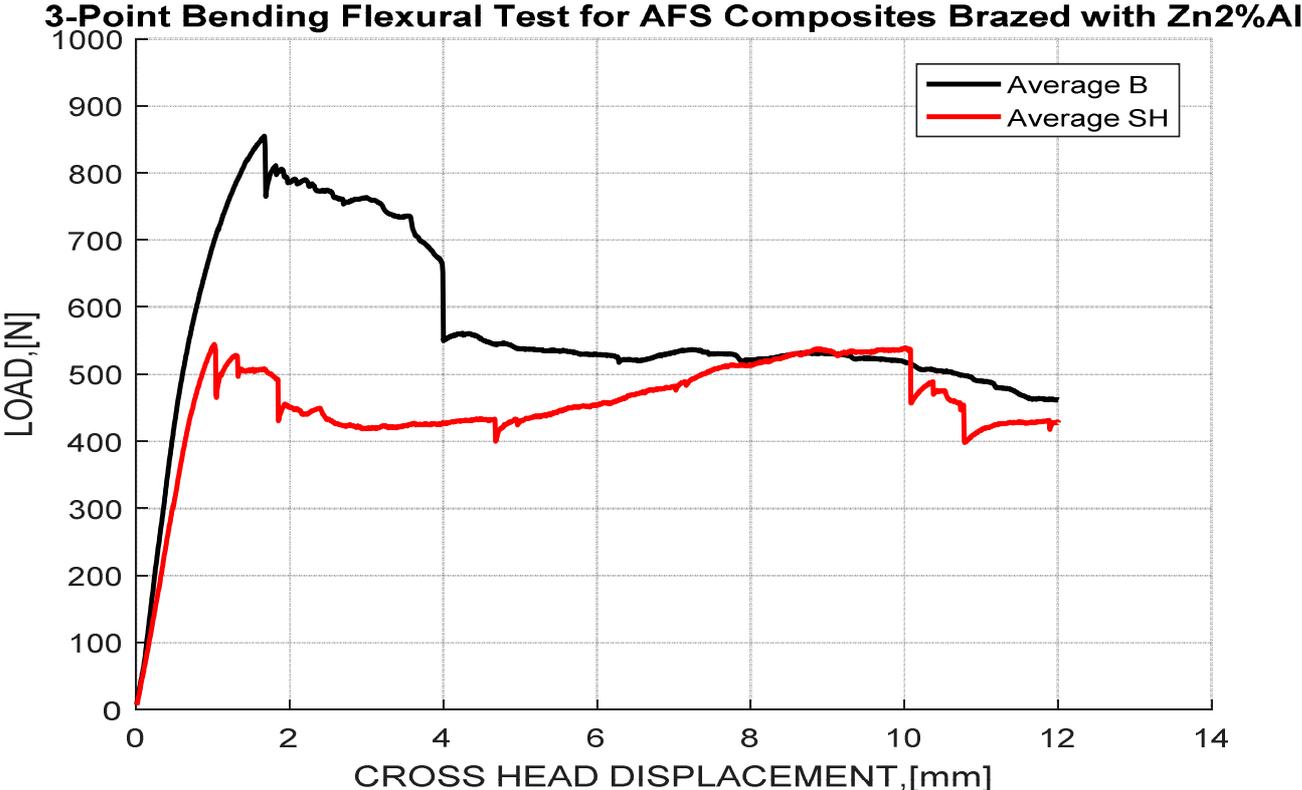


Figure 102: Average results of brazed and solution heat treated AFS composite

### Elastic Modulus

The results of elastic modulus was obtained from 3 point bending flexural test, also physical properties such as dimension, weight for each AFS composites Zn2%Al are reported below;

Table 22: Elastic modulus and physical properties of AFS composite brazed with Zn2%Al

Specimen	Length (mm)	Width (mm)	Thickness (mm)	Weight (g)	E-Mod (MPa)
B1	62.5	22.7	11.3	16.43	997
B5	61.7	21.9	11.17	16.59	1187
B6	63.6	22.4	10.5	16.75	721
SH2	61.9	23.1	11.1	17.55	725
SH3	61.3	22.9	11	17.88	768
SH4	61.3	23.3	11.5	16.59	549

The average modulus values of brazed and solution heat treated AFS composites were reported in the figure 103 and again brazed AFS composites have got higher modulus than solution heat treated ones. That means they have more resistance to being deformed elastically. When the modulus were compared between AFS composites brazed with pure Zn and Zn2%Al, it was seen that the values of modulus were higher when Zn2%Al was used as brazing material. The increase of modulus for only brazed AFS composite was 48% when Zn2%Al was used instead of pure Zn. Meanwhile, increase of modulus for solution heat treated AFS composite was 113% when Zn2%Al was used instead of pure Zn. However, solution heat treatment was not good approach for AFS composites in terms of maximum load and strength.

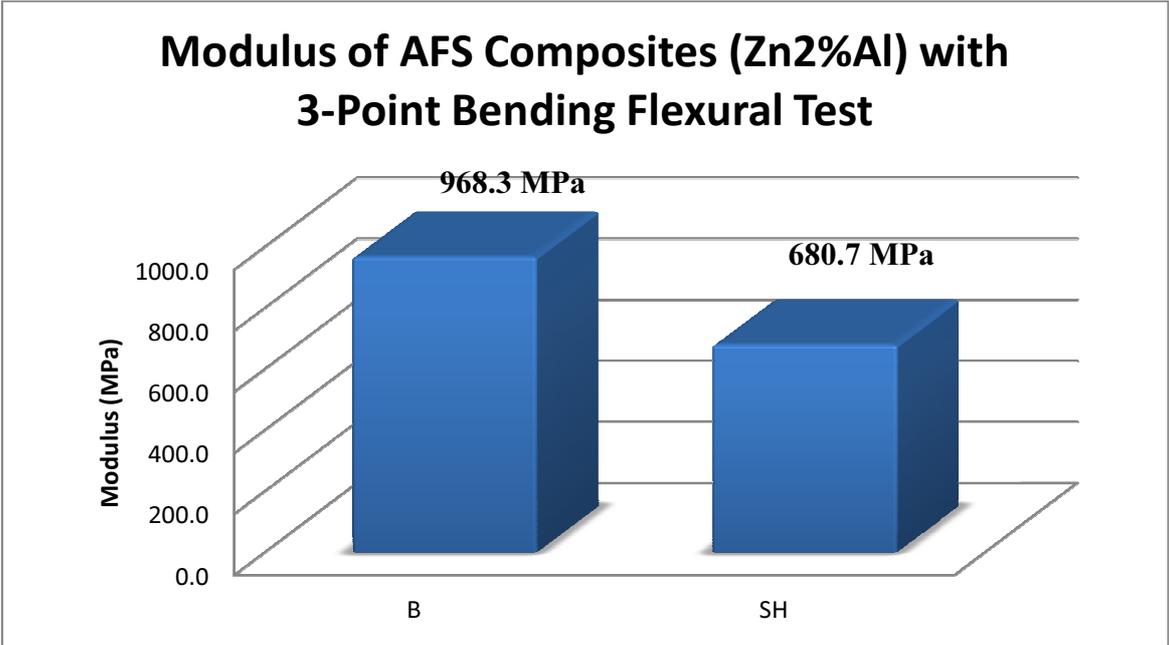


Figure 103: Modulus of AFS composites brazed Zn2%Al with 3 point bending flexural test

### 4.3.2 Scanning Electron Microscopy (SEM)

Microstructures of the joining seam between Al-foam and Al plate, also the area of Al-foam and Al plate were analyzed with different magnifications by SEM for brazed and solution heat treated AFS composites.

- **Brazed AFS composites**

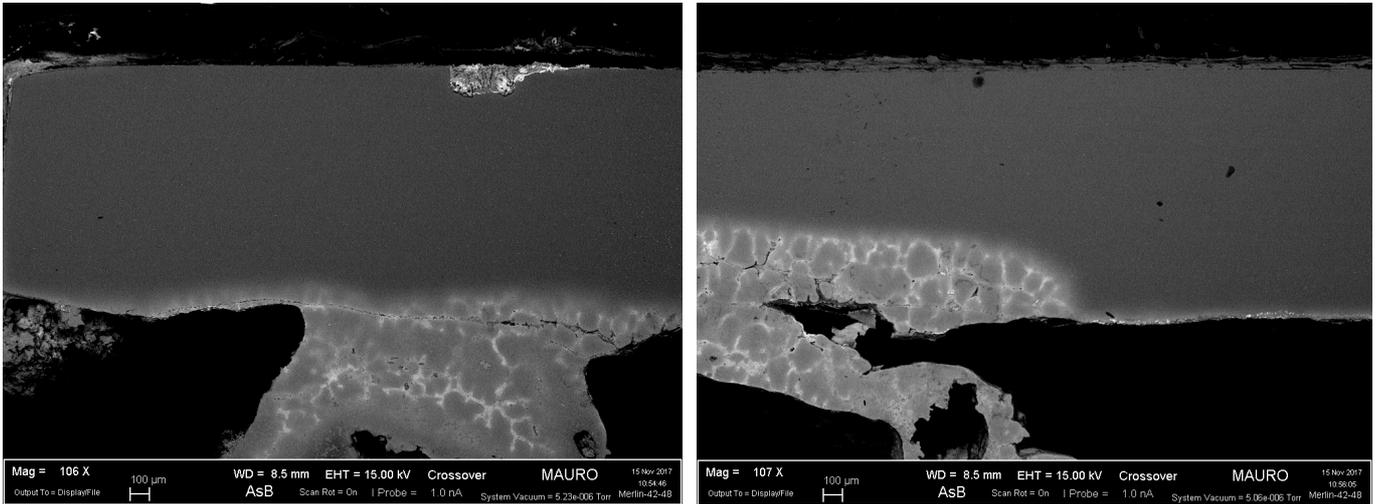


Figure 104: Microstructures of brazed AFS composites on joint seams, magnification 106X (on the left), 107X (on the right) 100 $\mu$ m

Joining seam line between Al plate and Al-foam is shown in the figure 104. The joining material Zn2%Al was melted during the brazing joining treatment and it was diffused between Al sheet and Al-foam. Also, dendrite structure was witnessed in the Al-foam region.

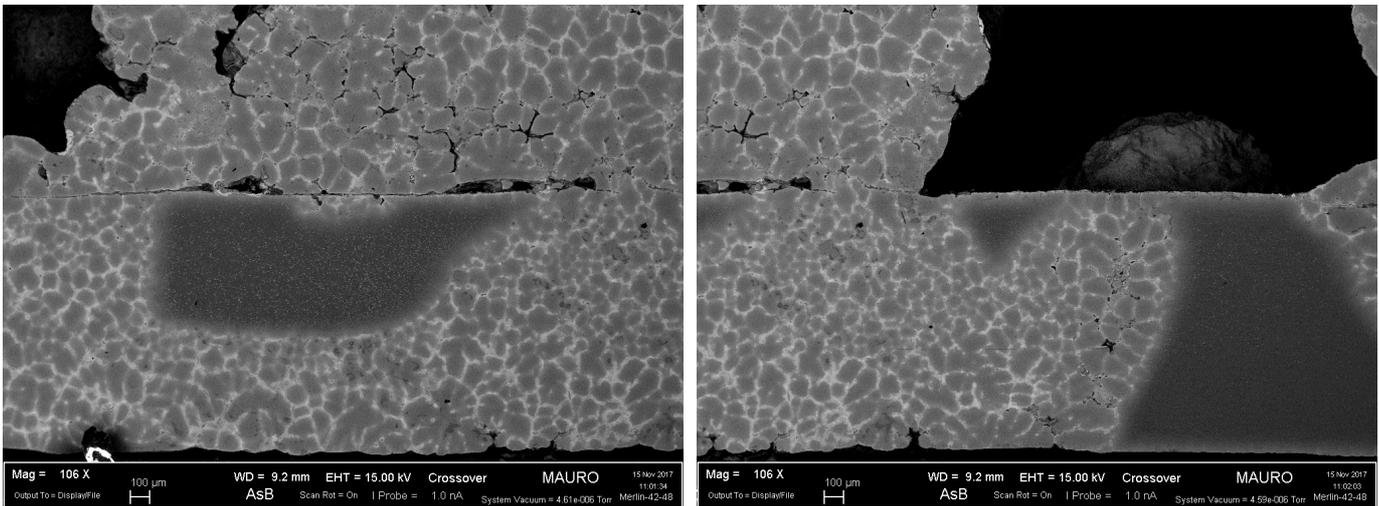


Figure 105: Microstructures of brazed AFS composites on joint seams, magnification 106X 100 $\mu$ m

The joining melt induced the diffusion between Al sheet and Al-foam and subsequently melt them. Following to the cooling, new liquid solution solidifies and create a microstructure that contains some amount of porosity mostly on the area close to the joining seam, shown in the figure 105, the porosity in the area close to the joining seam in the Al-foam region.

When the magnification was increased to 2.50KX, different phases were obtained in the microstructure of joint seam, shown in the figure 106.

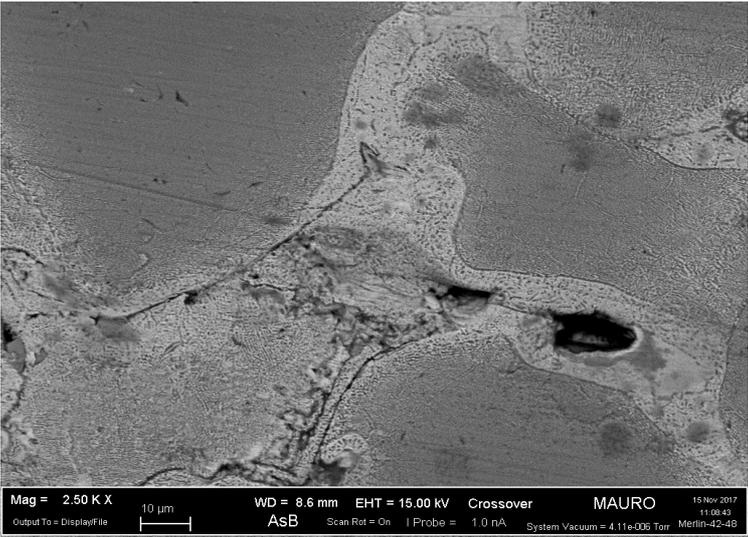


Figure 106: Microstructures of brazed AFS composites on joint seams, magnification 2.50KX 10µm

- **Solution heat treated AFS composite**

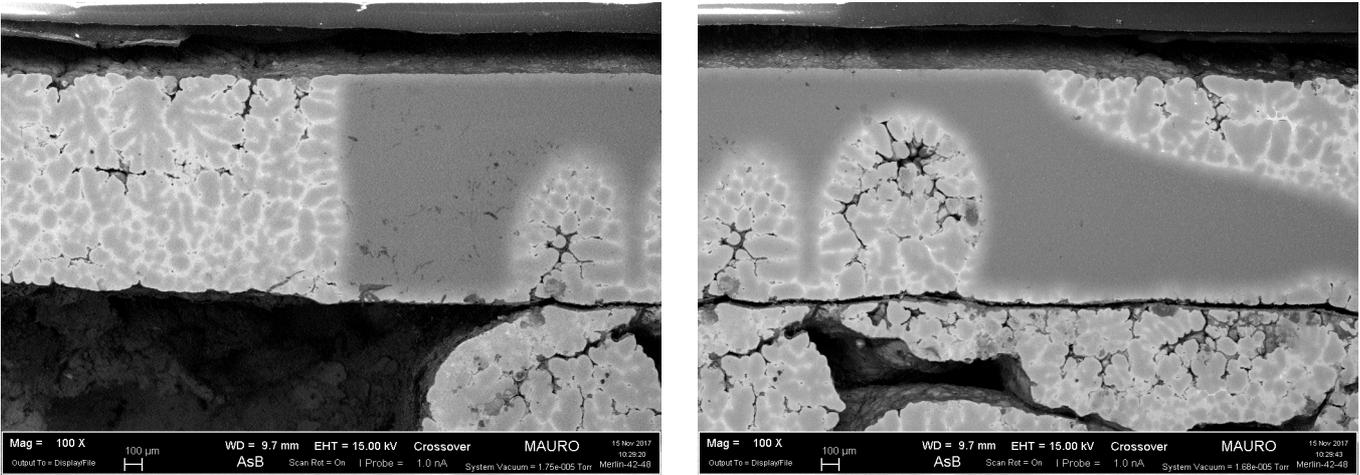


Figure 107: Microstructures of solution heat treated AFS composites on joint seams, magnification 100X 100µm

The joining seam between Al-foam and Al sheet again is shown in the figure 107 for solution heat treated AFS composites. In this group, porosity was observed not only on the Al-foam also on the Al sheet region. When the solution heat treatment was applied to the brazed AFS composites, the reason of the decrease of the strength and maximum load capacity on the 3 point bending test could be explained with existence of porosity microstructures both in the Al sheet and Al-foam. Whereas, porosity was observed only Al-foam region for brazed AFS composites. Because of that reason, solution heat treated AFS composites had lower values of strength and maximum load capacity on 3 point bending test.

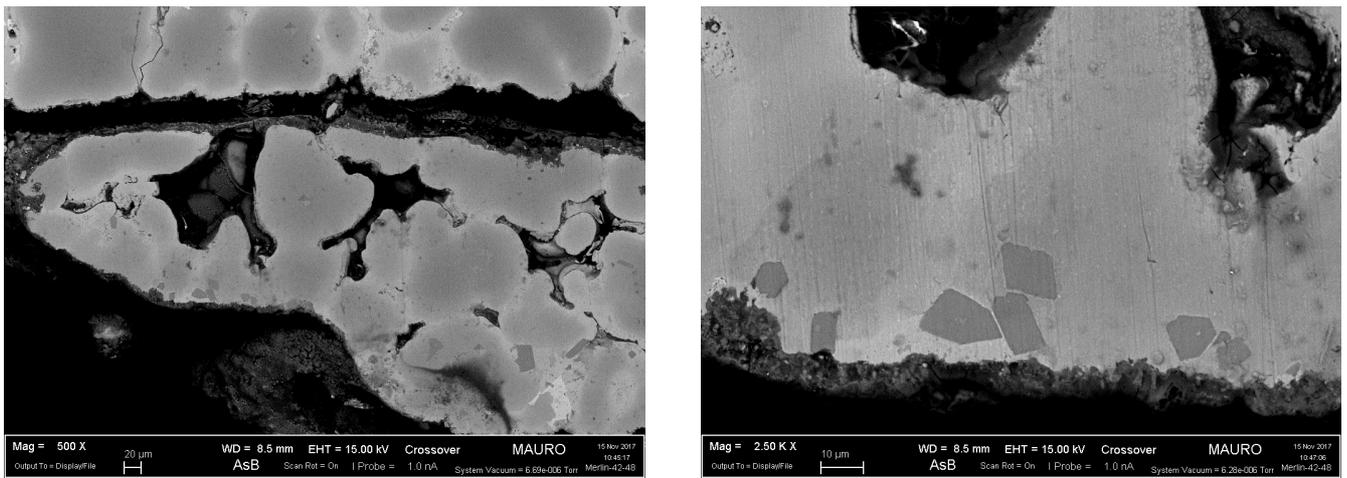


Figure 108: Microstructures of solution heat treated AFS composites on joint seams, magnification 500X 20µm (on the left), 2.50 KX 10µm (on the right)

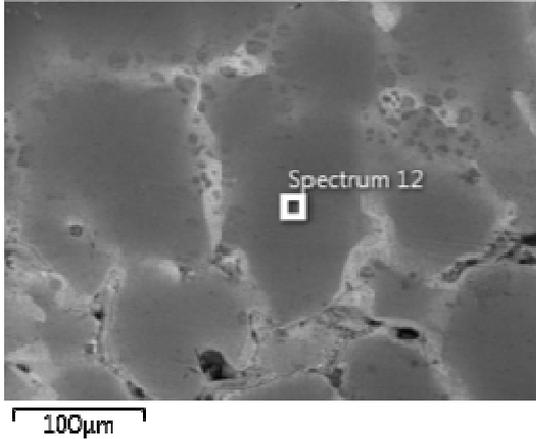
When the magnification was increased from 100X to 500X, shown in the figure 108 the left side, the porosity and voids which appeared in the joining seam of Al-foam side region formed during the solidification of the joint. Second phase was observed in the joint seam of Al-foam region when the magnification was increased to 2.50KX, in the figure 108 right side.

### 4.3.3 Energy Dispersive Spectrometry (EDS)

As following to the SEM images, EDS analyzes, for point or area, were conducted for AFS composite brazed with Zn2%Al and solution heat treated ones.

- **Brazed AFS Composites**

**Electron Image 4**

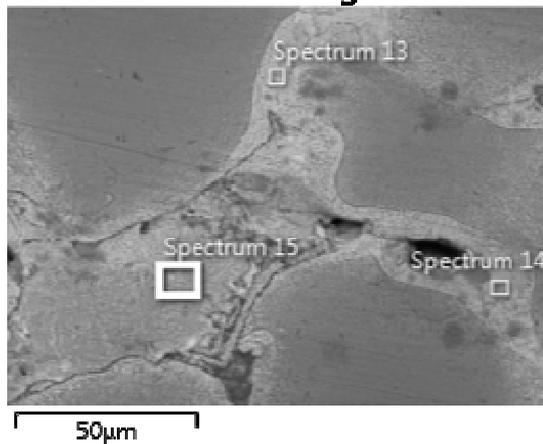


<b>Spectrum 12</b>			
Element	Wt%	Wt% Sigma	Atomic %
Al	67.49	0.31	83.41
Zn	32.51	0.31	16.59
Total:	100.00		100.00

Figure 109: Electron image, chemical structure of brazed AFS composite

Selected position in the matrix of brazed AFS composite, spectrum 12 is shown in the electron image 4, figure 109 and chemical analyze of this position is reported in the table. It is observed that the main alloying elements Al with 67.49% and Zn with 32.51% of the matrix structure of brazed AFS composite.

**Electron Image 5**



<b>Spectrum 13</b>		
Element	Wt%	Wt% Sigma
Al	24.34	0.34
Zn	75.66	0.34
Total:	100.00	

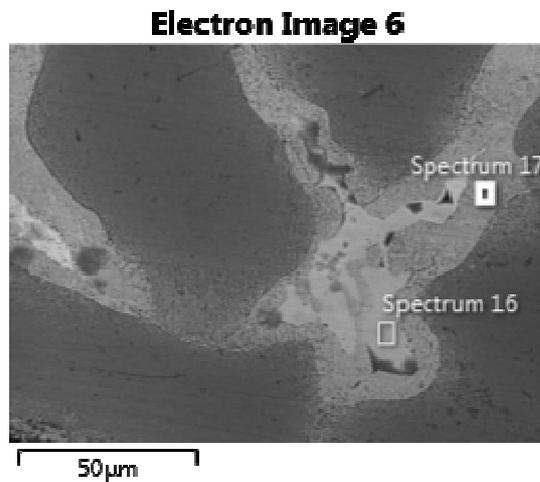
<b>Spectrum 14</b>			
Element	Wt%	Wt% Sigma	Atomic %
O	20.74	0.30	46.91
Mg	6.93	0.21	10.31
Al	3.46	0.14	4.64
Zn	68.87	0.34	38.13
Total:	100.00		100.00

Figure 110: Electron image, chemical structure of brazed AFS composite

The position of analysis in the diffusion zone of brazed AFS composite, spectrum 13, 14, 15 are shown in the electron image 5, figure 110 and chemical analyzes of these points are reported on the table. It was seen high amount of Zn around

<b>Spectrum 15</b>			
Element	Wt%	Wt% Sigma	Atomic %
Al	40.28	0.29	62.04
Zn	59.72	0.29	37.96
Total:	100.00		100.00

75% and the second main element was Al in the diffusion zone. Also, there was second phase in spectrum 14 that It was seen the amount of Mg around 7%. The Oxygen existence might be the reason of the small water drop on this area.



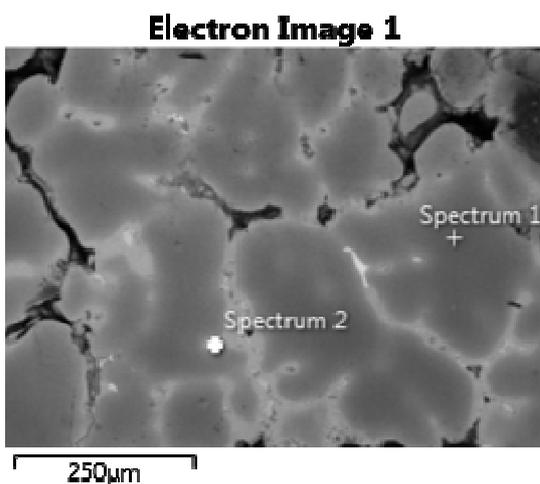
<b>Spectrum 16</b>			
Element	Wt%	Wt% Sigma	Atomic %
Mg	14.82	0.34	31.44
Al	1.22	0.16	2.34
Zn	83.96	0.37	66.23
Total:	100.00		100.00

<b>Spectrum 17</b>			
Element	Wt%	Wt% Sigma	Atomic %
Al	21.87	0.30	40.41
Zn	78.13	0.30	59.59
Total:	100.00		100.00

Figure 111: Electron image, chemical structure of brazed AFS composite

Another positions of analysis in the diffusion zone of brazed AFS composite, spectrum 16, 17 are shown in the electron image 6, figure 111 and chemical analyzes of these points are reported in the table. It was again seen high amount of Zn around 78% and the second main element was Al in the diffusion zone. There was also second phase precipitation in spectrum 16 and the amount of Mg was around 15%.

- **Solution Heat Treated AFS Composites**



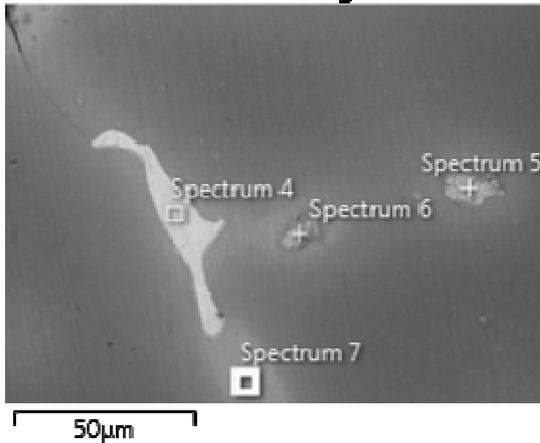
<b>Spectrum 1</b>			
Element	Wt%	Wt% Sigma	Atomic %
Al	69.26	0.35	84.52
Zn	30.74	0.35	15.48
Total:	100.00		100.00

<b>Spectrum 2</b>			
Element	Wt%	Wt% Sigma	Atomic %
Mg	0.59	0.11	1.07
Al	32.82	0.22	53.84
Zn	66.59	0.23	45.09
Total:	100.00		100.00

Figure 112: Electron image, chemical structure of solution heat treated AFS composite

The position of analysis for solution heat treated AFS composite, spectrum 1, 2 are shown in the electron image 1, figure 112 and chemical analyzes of these points are reported in the table. Spectrum 1 was in the matrix form of the solution heat treated AFS composite whereas Spectrum 2 was in the diffusion zone with high amount of Zn around 67%.

**Electron Image 2**



<b>Spectrum 4</b>			
Element	Wt%	Wt% Sigma	Atomic %
Al	2.90	0.18	6.74
Zn	97.10	0.18	93.26
Total:	100.00		100.00

<b>Spectrum 5</b>			
Element	Wt%	Wt% Sigma	Atomic %
Mg	10.28	0.26	18.67
Al	21.52	0.28	35.21
Fe	0.62	0.20	0.49
Zn	67.58	0.36	45.64
Total:	100.00		100.00

<b>Spectrum 6</b>			
Element	Wt%	Wt% Sigma	Atomic %
O	12.72	0.20	30.54
Mg	9.31	0.17	14.70
Al	10.68	0.15	15.21
Zn	67.29	0.25	39.55
Total:	100.00		100.00

<b>Spectrum 7</b>			
Element	Wt%	Wt% Sigma	Atomic %
Al	35.17	0.34	56.79
Zn	64.83	0.34	43.21
Total:	100.00		100.00

Figure 113: Electron image, chemical structure of solution heat treated AFS composite

The position of analysis for solution heat treated AFS composite, spectrum 4, 5, 6, 7 are shown in the electron image 2, figure 113 and chemical analyzes of these positions are reported in the table. Spectrum 5 and 6 were in the second phase with Mg around 10% whereas Spectrum 4 was in the highly diffused precipitation with high amount of Zn around 97%. However, Spectrum 7 was also in the diffusion zone with high amount of Zn around 65% as shown before with Spectrum 2.

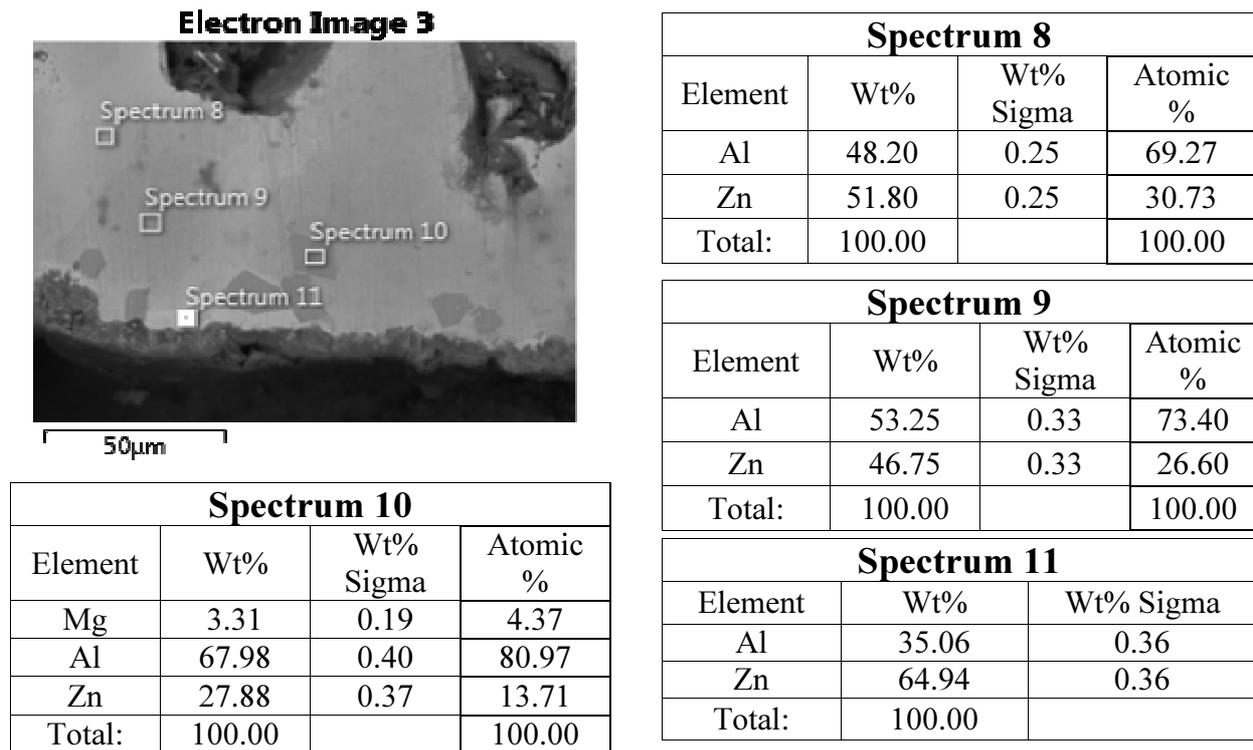


Figure 114: Electron image, chemical structure of solution heat treated AFS composite

The position of analysis for solution heat treated AFS composite, spectrum 8, 9, 10, 11 are shown in the electron image 3, figure 114 and chemical analyzes of these points are reported in the table. Spectrum 10 was in the second phase with Mg around 3% whereas Spectrum 8, 9, 11 were in the diffusion zone, Spectrum 11 with high amount of Zn around 65% as shown before with Spectrum 2 and 7.

### 4.3.4 Micro Hardness Test

The Vickers micro hardness test were performed for brazed AFS composite with Zn2%Al and solution heat treated AFS composite.

- **Brazed AFS composite**
  - Al-7046 plate edge of the joining seam of the AFS composite



Figure 115: Micro Hardness of Al-plate edge of the joining seam of AFS composite

- Diffusion zone in the joining seam of the AFS composite

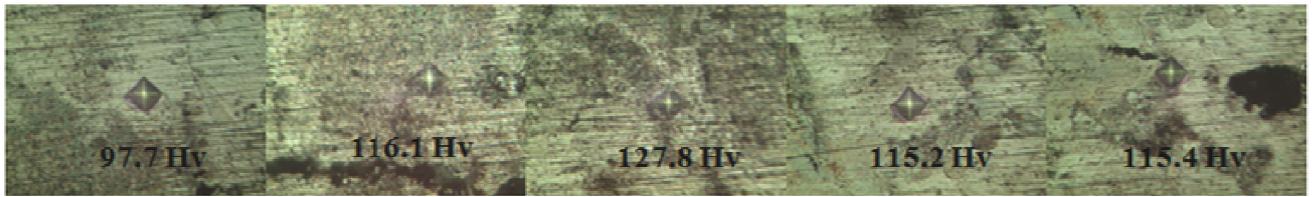


Figure 116: Hardness of diffusion zone in the joining seam of AFS composite

Hardness was increased on the diffusion zone of the joining seam of AFS composite.

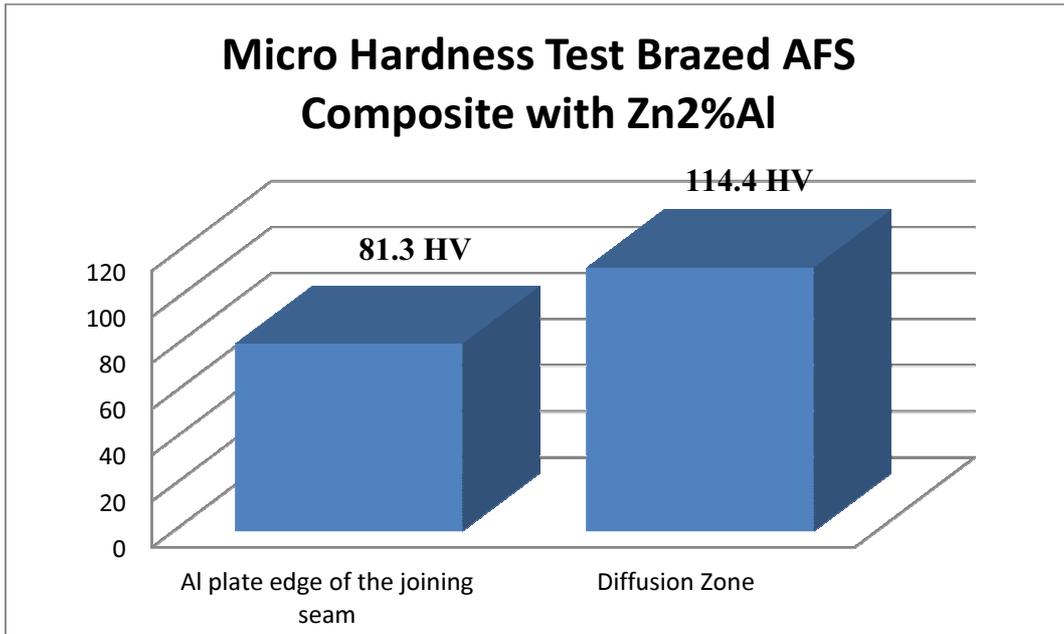


Figure 117: Micro hardness average results of brazed AFS composite with Zn2%Al

- **Solution Heat Treated AFS composite**

- Al-7046 plate edge of the joining seam of AFS composite



Figure 118: Micro Hardness of Al-plate edge of the joining seam of AFS composite

- Al-Foam on the joining seam of AFS composite



Figure 119: Micro Hardness of Al-Foam on the joining seam of AFS composite

- Diffusion zone in the joining seam of AFS composite

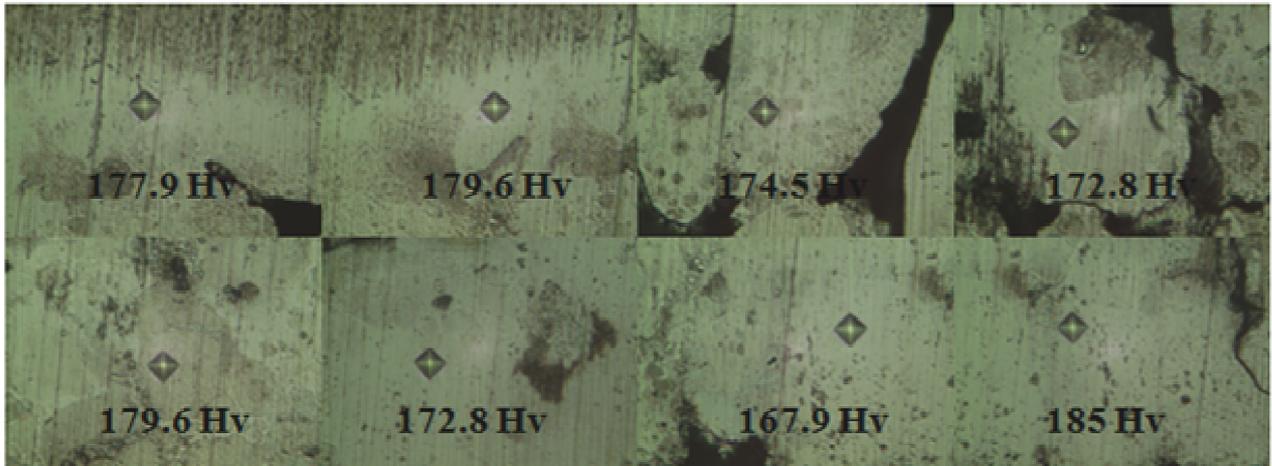


Figure 120: Micro Hardness of diffusion zone in the joining seam of AFS composite

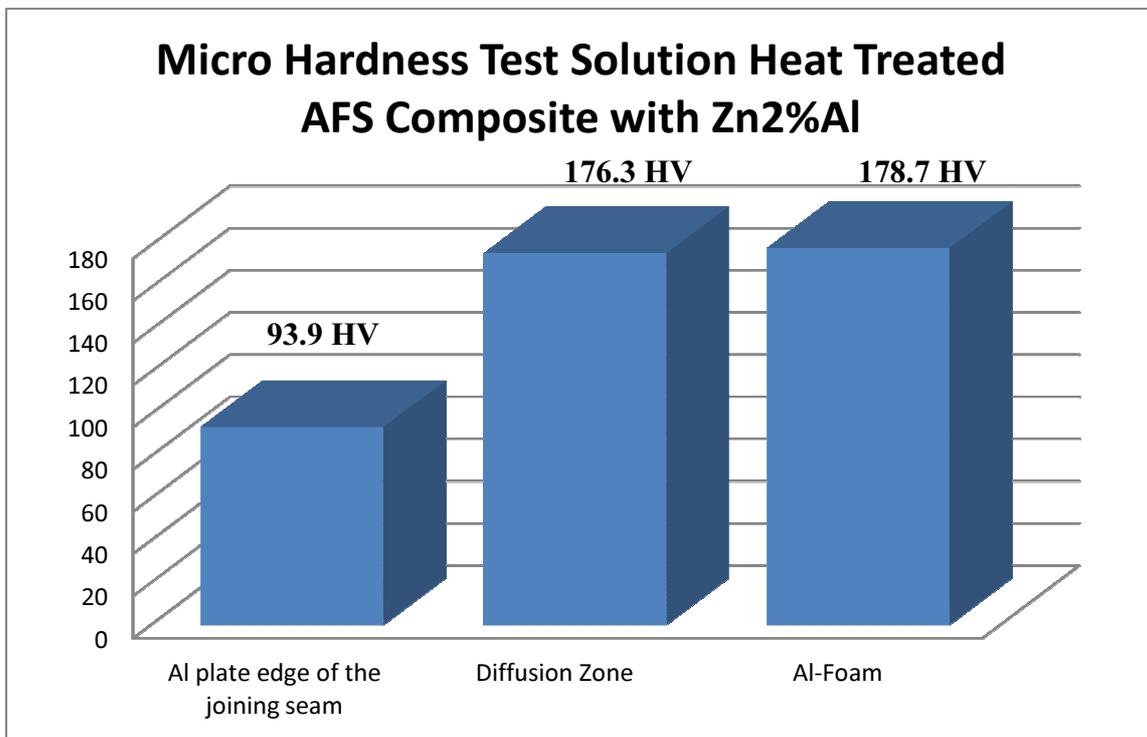


Figure 121: Micro hardness average results of solution heat treated AFS composite with Zn2%Al

As reported in the figure 121, micro hardness average value was increased in the diffusion zone again. Comparing to the brazed AFS composite, it was observed the increase of hardness from 83.1 HV to 93.9 HV in Al plate edge of the joining seam and from 114.4 HV to 176.3 HV in diffusion zone with the solution heat treatment.

## 4.4 2nd Batch of Al-7046 Alloys

### 4.4.1 Scanning Electron Microscopy (SEM)

SEM images were obtained with the different magnifications such as 1.00KX, 3.50KX and 10.00KX for as received, brazed heat treated and solution heat treated 2<sup>nd</sup> batch Al-7046 alloys.

- **1.00KX Magnification, 10 $\mu$ m**

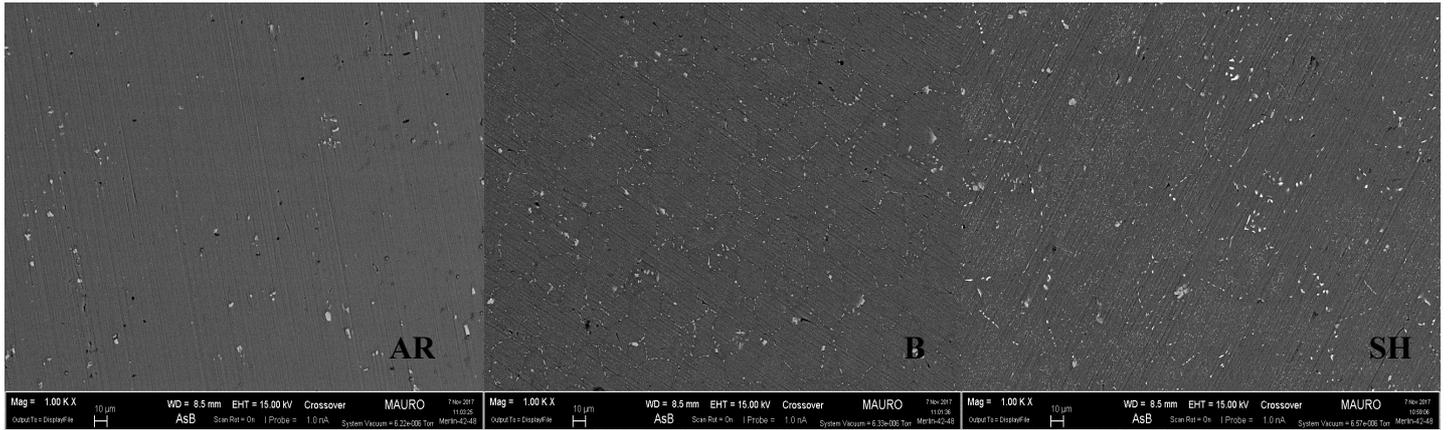


Figure 122: SEM microstructures as received (on the left), brazed heat treated (in the middle), solution heat treated (on the right)

- **3.50KX Magnification**

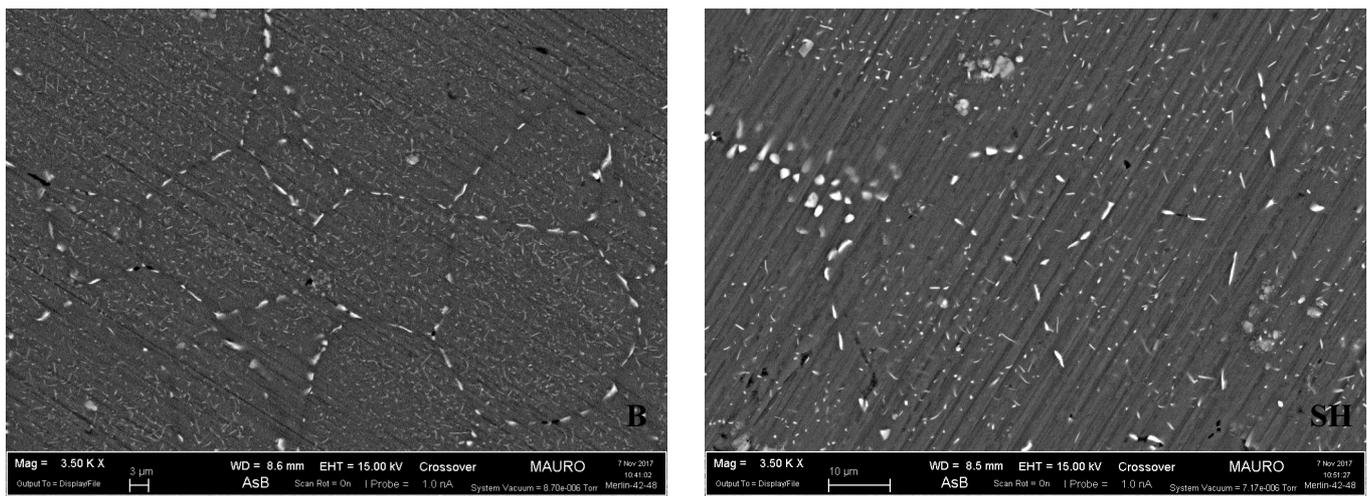


Figure 123: SEM microstructures brazed heat treated (on the left) 3 $\mu$ m, solution heat treated (on the right) 10 $\mu$ m

- **10KX Magnification, 1 $\mu$ m**

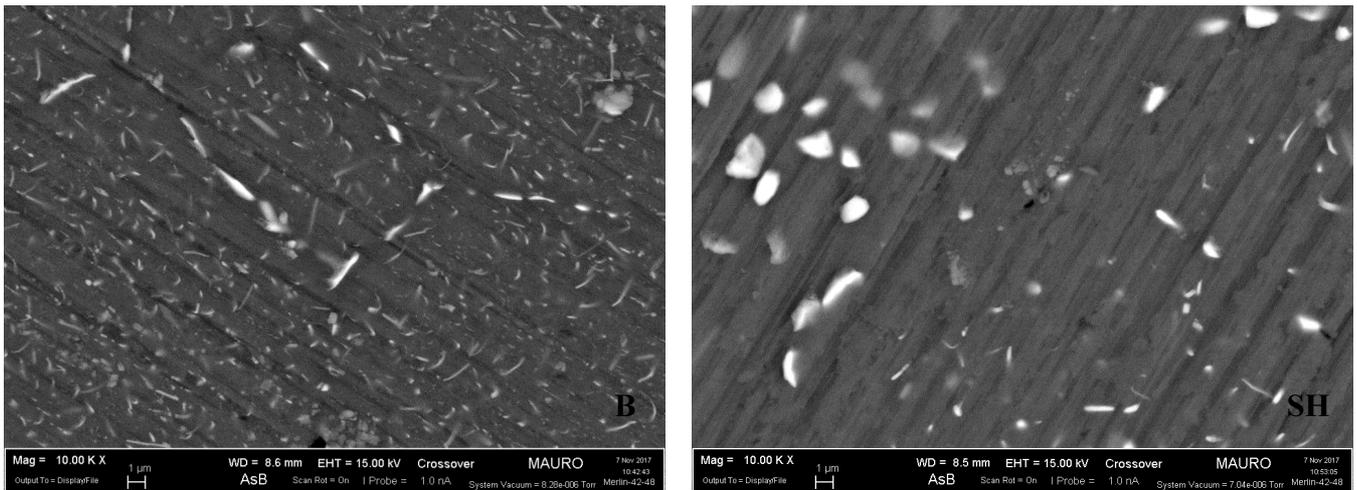


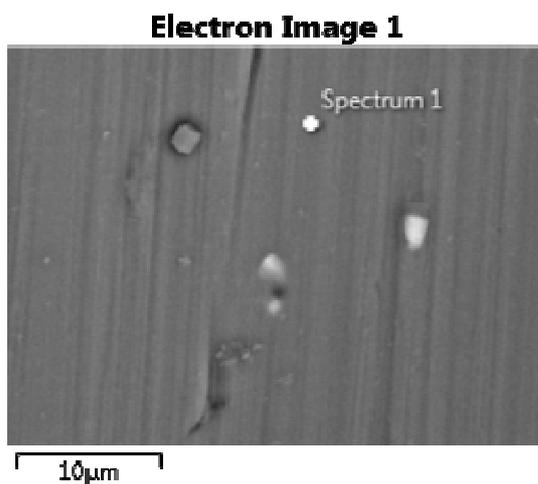
Figure 124: SEM microstructures brazed heat treated (on the left), solution heat treated (on the right)

In the SEM images of as received Al-7046 alloys, it is observed some small particles due to its own structure being alloy with different elements.

#### 4.4.2 Energy Dispersive Spectrometry (EDS)

As following to the SEM images, EDS analyzes, for position of the spectrum or area, were conducted for these 3 groups of 2<sup>nd</sup> batch Al-7046 alloys.

- **As received 2<sup>nd</sup> batch Al-7046**

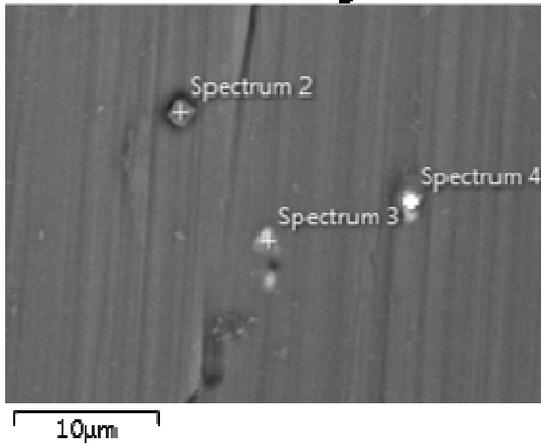


Spectrum 1			
Element	Wt%	Wt% Sigma	Atomic %
Mg	1.95	0.09	2.26
Al	90.81	0.20	94.63
Zn	7.24	0.18	3.11
Total:	100.00		100.00

Figure 125: Electron image and its chemical structure

The area of the electron image which obtained from SEM was selected for chemical analyze and the result is reported in the table. The main alloying element is Al and Zn is the second following main alloying element.

### Electron Image 2



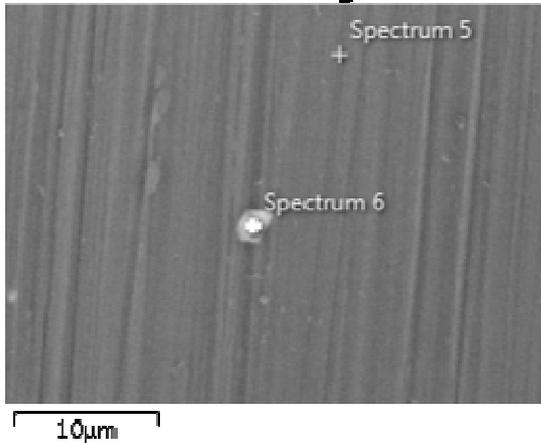
Spectrum 3			
Element	Wt%	Wt% Sigma	Atomic %
Al	77.72	0.57	88.19
Fe	17.28	0.52	9.47
Zn	5.00	0.35	2.34
Total:	100.00		100.00

Spectrum 4		
Element	Wt%	Wt% Sigma
Mg	1.68	0.09
Al	88.84	0.30
Fe	3.48	0.24
Zn	6.00	0.20
Total:	100.00	

Figure 126: Electron images and their chemical structures

The positions of the analysis, spectrum 3 and spectrum 4, are shown in the figure 126 and chemical analyzes of these points are reported in the tables. There is high amount of Fe in the as received Al-7046 alloy and it might be due to precipitation in the main alloy. However, this high amount of Fe became less active on the position of spectrum 4.

### Electron Image 3



Spectrum 5		
Element	Wt%	Wt% Sigma
Mg	1.89	0.13
Al	90.56	0.30
Zn	7.55	0.28
Total:	100.00	

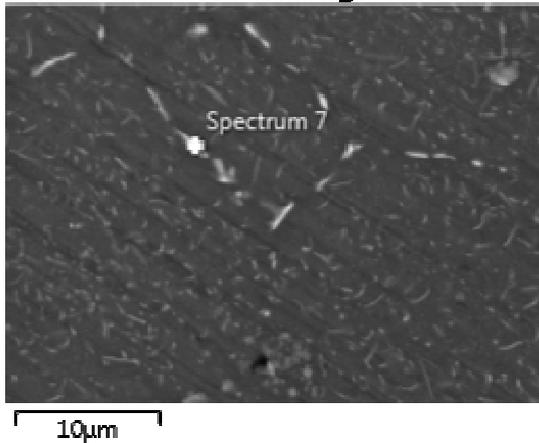
Spectrum 6		
Element	Wt%	Wt% Sigma
Al	81.19	0.34
Fe	13.29	0.31
Zn	5.52	0.21
Total:	100.00	

Figure 127: Electron images and their chemical structures

The positions of the analysis, spectrum 5 and spectrum 6, are shown in the figure 127 and chemical analyzes of these points are reported in the tables. The position of spectrum 5 was formed in the base structure of as received Al-7046 alloy and the position of spectrum 6 was precipitation in the main alloy as shown previously above in the position of spectrum 3.

- Brazed heat treated 2<sup>nd</sup> batch Al-7046

**Electron Image 4**

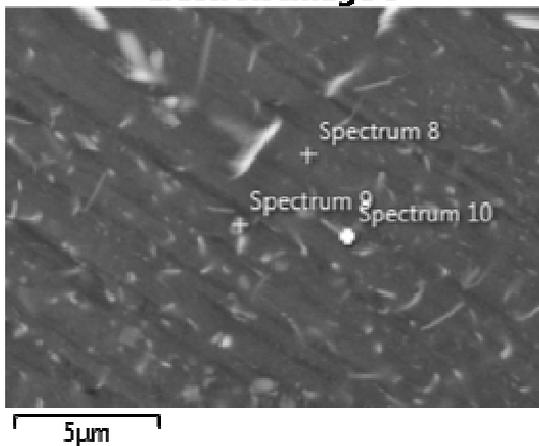


<b>Spectrum 7</b>		
Element	Wt%	Wt% Sigma
Mg	1.94	0.08
Al	87.88	0.19
Zn	10.18	0.17
Total:	100.00	

Figure 128: Electron image and its chemical structures

Selected position in the coarse grain boundary, spectrum 7 is shown in the figure 128 and chemical analyze of this position is reported in the table. It is observed that the amount of Zn in the alloy structure became more evident with brazing heat treatment in the formed grain boundary.

**Electron Image 5**



<b>Spectrum 8</b>		
Element	Wt%	Wt% Sigma
Mg	1.51	0.10
Al	91.74	0.23
Zn	6.75	0.21
Total:	100.00	

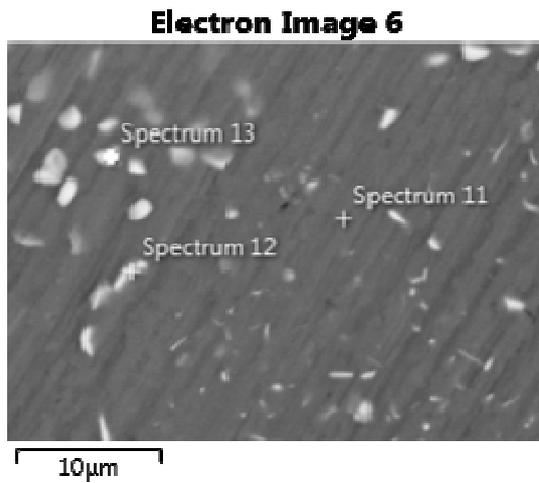
<b>Spectrum 9</b>		
Element	Wt%	Wt% Sigma
Mg	1.80	0.09
Al	89.63	0.20
Zn	8.57	0.19
Total:	100.00	

<b>Spectrum 10</b>		
Element	Wt%	Wt% Sigma
Mg	1.37	0.09
Al	91.90	0.22
Zn	6.72	0.21
Total:	100.00	

Figure 129: Electron images and their chemical structures

Grains and their boundaries are shown in the electron image 5. Selected positions, spectrum 8,9,10 are shown in the figure 129 and chemical analyzes of these positions are reported in the tables. The position of spectrum 8 and 10 were formed in the base structure of brazed heat treated Al-7046 alloy and the position of spectrum 9 was in the grain with high amount of Zn.

- Solution heat treated 2<sup>nd</sup> batch Al-7046



<b>Spectrum 11</b>		
Element	Wt%	Wt% Sigma
Mg	1.21	0.09
Al	92.80	0.20
Zn	5.99	0.19
Total:	100.00	

<b>Spectrum 12</b>		
Element	Wt%	Wt% Sigma
Mg	3.44	0.16
Al	80.05	0.35
Zn	16.51	0.33
Total:	100.00	

<b>Spectrum 13</b>		
Element	Wt%	Wt% Sigma
Mg	2.05	0.12
Al	86.31	0.29
Zn	11.64	0.28
Total:	100.00	

Figure 130: Electron images and their chemical structures

Grains of the 2<sup>nd</sup> batch solution heat treated Al-7046 alloy are shown in the electron image 6. Selected positions, spectrum 11, 12, 13 are shown in the figure 130 and chemical analyzes of these positions are reported in the tables. The position of spectrum 11 was formed in the base structure of solution heat treated Al-7046 alloy and the position of spectrum 11 and 12 were in the grain with high amount of Zn.

Compared with the base structure of brazed heat treated Al-7046 alloy, it was observed the appearance of Al element became more active and Zn element became less active in the alloy structure.

### 4.4.3 Micro Hardness Test

The Vickers micro hardness test were performed for as received (AR), brazed heat treated (B) and solution heat treated (SH) 2<sup>nd</sup> batch Al-7046 alloys, shown in the figure 131.

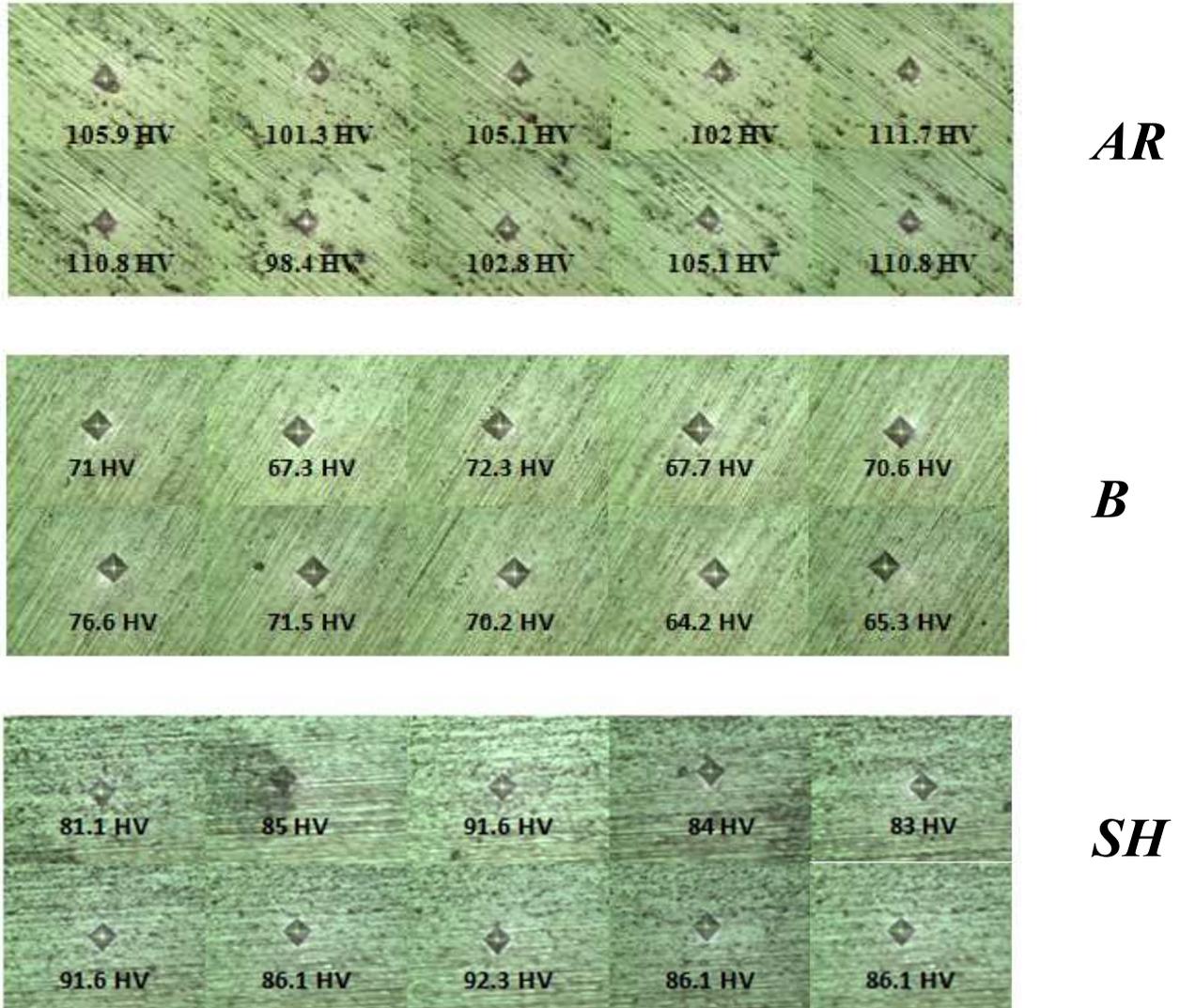


Figure 131: Micro hardness test results for 2<sup>nd</sup> batch Al-7046 alloys

As reported the average hardness values in the figure 132, the maximum hardness was obtain for as received 2<sup>nd</sup> batch Al-7046 alloy then solution heat treated and the minimum hardness for brazed heat treated 2<sup>nd</sup> batch Al-7046 alloy. It was expected to have decrease of strength and hardness after brazing heat treatment due to long time of cooling and microstructures that grain boundaries became coarser and bigger.

As following to the solution heat treatment after brazing heat treatment, good amount of hardness was recovered. Thus, solution heat treatment was beneficial for recovering the micro hardness of the Al-7046 alloys after brazing heat treatment.

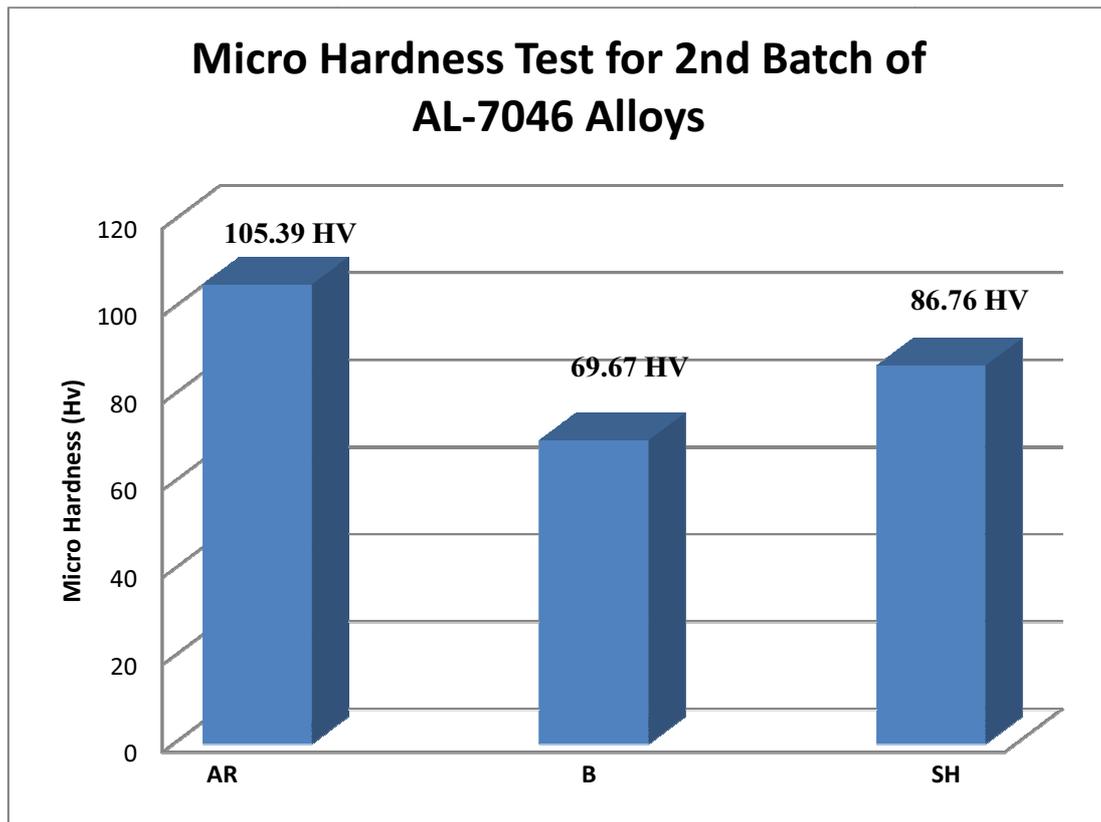


Figure 132: Micro hardness test results for 2<sup>nd</sup> batch Al-7046 alloys and their average hardness values

When the hardness data of 1<sup>st</sup> batch of Al-7046 alloys were checked, it was observed the difference on hardness value of brazed heat treated sheets was 78.6 HV for 1<sup>st</sup> batch whereas 69.67HV for 2<sup>nd</sup> batch. The reason of this decrease of around 10HV would be explained with applying higher temperature 500°C as brazing temperature meanwhile brazing temperature was 450°C for the 1<sup>st</sup> batch. This higher temperature made the microstructure coarser than the 1<sup>st</sup> batch and let the samples have less hardness.

The hardness is decreased 33.9% when brazing heat treatment was conducted for 2<sup>nd</sup> batch. Then, when the solution heat treatment was applied to the brazed 2<sup>nd</sup> batch Al-7046 alloys, the hardness of the brazed sheets was increased 24.5%.

The hardness of 1<sup>st</sup> batch as received Al-7046 is decreased 26.55% when brazing heat treatment was conducted, this value was 33.9% for 2<sup>nd</sup> batch, thus it could be said that the reason was again brazing temperature. When the solution heat treatment was applied for the brazed 1<sup>st</sup> batch Al-7046 alloys, the hardness of the brazed sheets was increased 28.4%.

#### 4.4.4 3-Point Bending Test

3 point bending flexural test were performed for 2<sup>nd</sup> batch Al-7046 alloys to the as received (AR), brazed heat treated (B) and solution heat treated (SH) specimens as following;

- Specimen Size

Table 23: Specimen features of 2<sup>nd</sup> batch of Al-7046 alloys

<b>Specimen</b>	<b>Length (mm)</b>	<b>Width (mm)</b>	<b>Thickness (mm)</b>
AR1	58.4	17.7	1.3
AR2	59.8	18.8	1.3
AR3	59.4	19.1	1.3
B1	60.4	17.9	1.3
B2	59.7	18.3	1.3
B3	60.4	18.5	1.3
SH1	55.8	20.5	1.3
SH2	57.6	19.7	1.3
SH3	59.4	19.3	1.3

- Span Length = 47 mm
- Loading Speed = 4mm/min
- Total 3 samples as received, 3 samples brazing heat treated, 3 samples brazing heat treated after solution heat treated

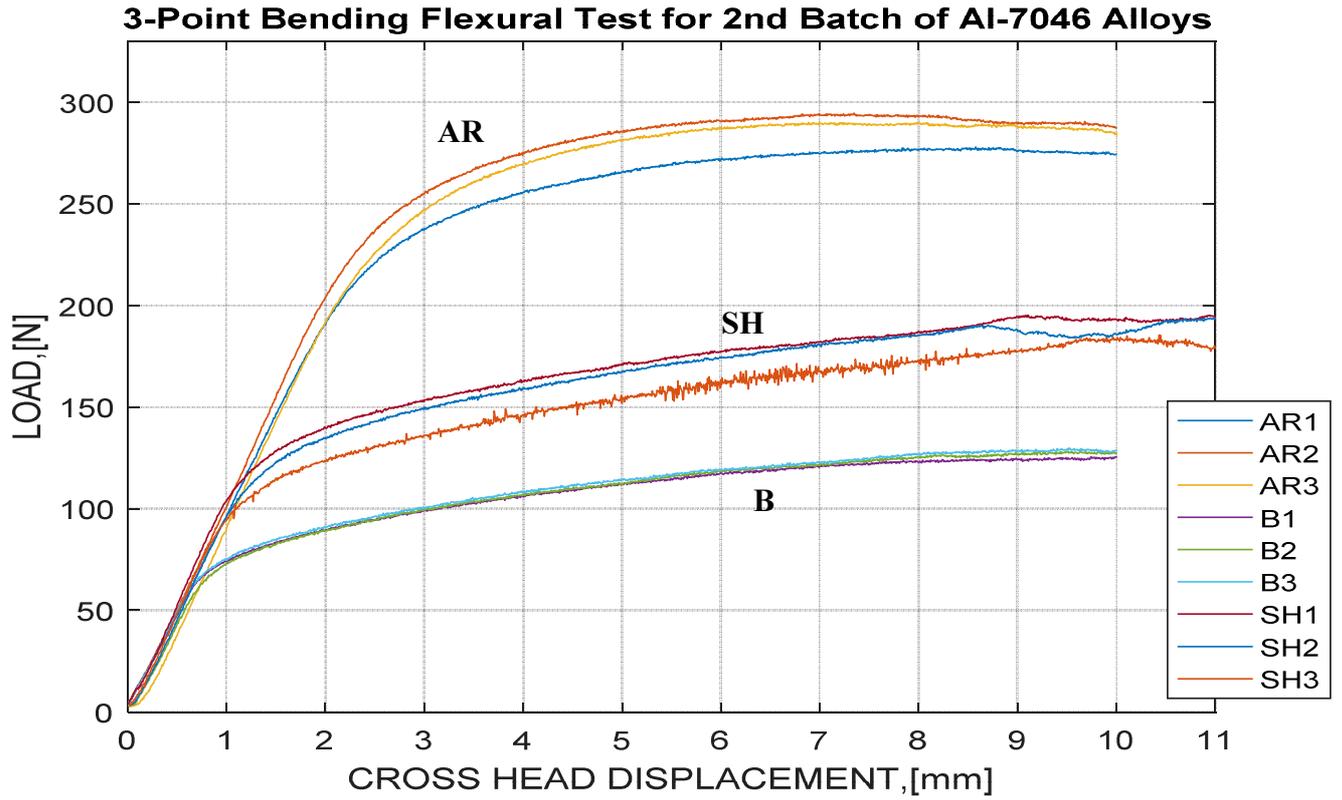


Figure 133: 3 point bending flexural test results for 2<sup>nd</sup> batch of Al-7046 alloys

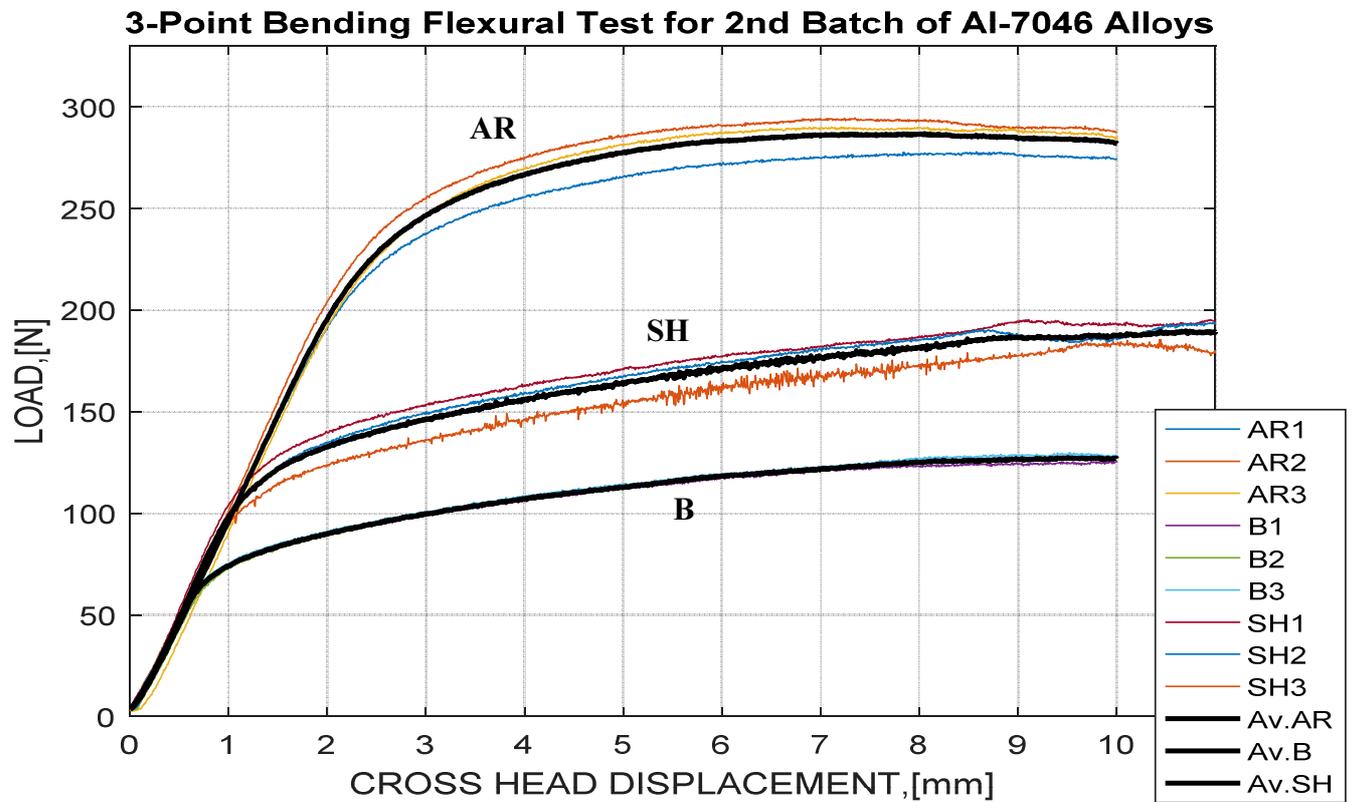


Figure 134: 3 point bending flexural test average values for 2<sup>nd</sup> batch of Al-7046 alloys

As reported in the figure 133 that the maximum strength was obtained for as received 2<sup>nd</sup> batch Al-7046 alloys, it was around again 280 N. Referring to the literature that there should be decrease of strength after brazing heat treatment, there was decrease of strength after brazing heat treatment around 54% due to long cooling time on the treatment and their microstructure became coarser after this treatment. Solution heat treatment was performed to recovery the strength and it is observed an increase of 46% of brazing heat treated strength.

When comparing the results of 2<sup>nd</sup> batch with 1<sup>st</sup> batch, the brazing heat treatment parameters for the 1<sup>st</sup> batch were 450°C brazing temperature, 10 minutes dwell time at maximum temperature, 12°C per minute heating rate and 10°C per minute cooling rate. Also, brazing heat treatments parameters for the 2<sup>nd</sup> batch were 500°C brazing temperature, 5 minutes dwell time at maximum temperature, 16.6°C per minute heating rate and 10°C per minute cooling rate. It was seen for the maximum load capacity of brazed 1<sup>st</sup> batch Al-7046 alloy was around 200N whereas maximum load capacity was dropped to 130N for brazed 2<sup>nd</sup> batch Al-7046 alloy due to higher brazing temperature.

The maximum load capacity was 220N for the solution heat treated 1<sup>st</sup> batch Al-7046 alloy and 10% load capacity was gained with solution heat treatment after brazing heat treatment. Whereas the maximum load capacity was around 190N for the solution heat treated 2<sup>nd</sup> batch Al-7046 alloy and 46% load capacity was gained. It could be said that solution heat treatment for the 2<sup>nd</sup> batch was more effective; however the decrease of load capacity after brazing heat treatment for 2<sup>nd</sup> batch was higher than the 1<sup>st</sup> batch.

Also, average curves of these 3 samples from each group is reported in the figure 134 show the benefit of solution heat treatment effected on the samples after brazing heat treatment. It was also seen the positive effects of solution heat treatment in terms of strength and mechanical properties as literature recommended.

For each sample, the other 3 point bending flexural test results such as elastic modulus and samples physical properties such as dimensions and weight are shown below.

Table 24: Elastic modulus and physical properties of 2<sup>nd</sup> batch Al-7046 alloy

<b>Specimen</b>	<b>Length (mm)</b>	<b>Width (mm)</b>	<b>Thickness (mm)</b>	<b>Weight (g)</b>	<b>E-Mod (GPa)</b>
AR1	58.4	17.7	1.3	3.64	64.616
AR2	59.8	18.8	1.3	3.95	64.069
AR3	59.4	19.1	1.3	4	64.724
B1	60.4	17.9	1.3	3.8	61.038
B2	59.7	18.3	1.3	3.84	62.995
B3	60.4	18.5	1.3	3.87	63.583
SH1	55.8	20.5	1.3	4.01	65.428
SH2	57.6	19.7	1.3	3.94	64.698
SH3	59.4	19.3	1.3	4.02	65.499

The average elastic modulus of solution heat treated 2<sup>nd</sup> batch Al-7046 alloys had the maximum modulus value in these 3 groups, shown in the figure 135. However, all the average elastic modulus values were close to each other.

Having not the same precise dimensions for the samples may be created this little fluctuation on elastic modulus in these 3 groups. There was 4% decrease of elastic modulus on brazed heat treated Al-7046 samples when compared with solution heat treated samples. It may said that from the average modulus data that solution heat treated 2<sup>nd</sup> batch Al-7046 alloys have more resistance to being deformed elastically when a stress is applied to it.

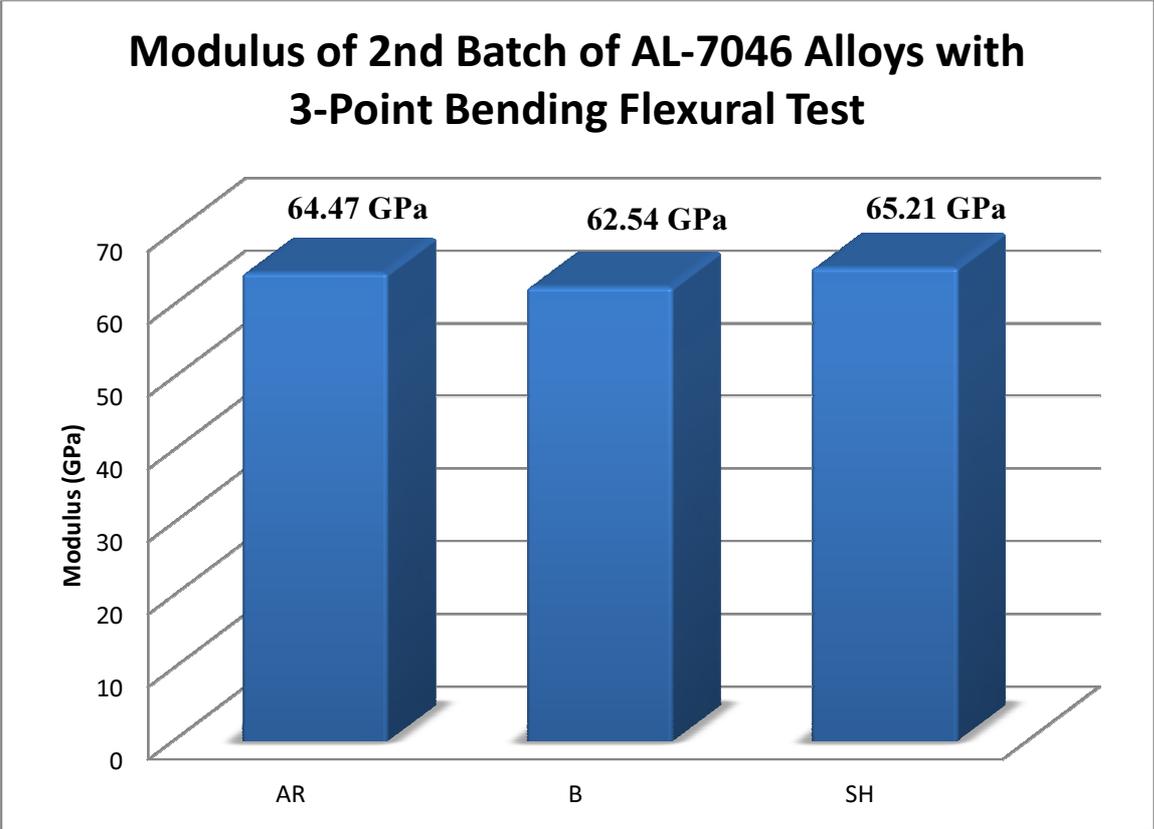


Figure 135: Modulus of 2<sup>nd</sup> batch Al-7046 alloys with 3 point bending flexural test

## 4.5 Aluminium Foam

### 4.5.1 Micro Hardness Test

Micro hardness test was conducted for as received aluminium foam, brazed heat treated aluminium foam and solution heat treated aluminium foam as following.

- **As Received Aluminium Foam**

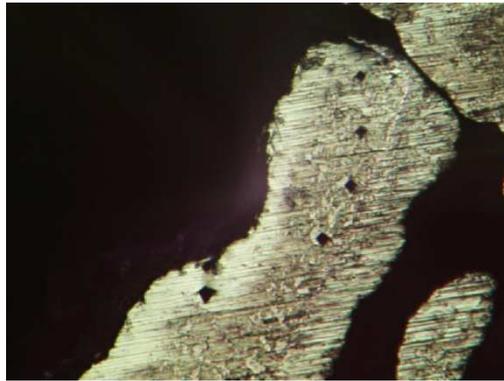


Figure 136: Micro hardness image of Al-Foam

There were different phases on the as received aluminium foam such as matrix form, second phase, and their average hardness value is in the following figure 137.

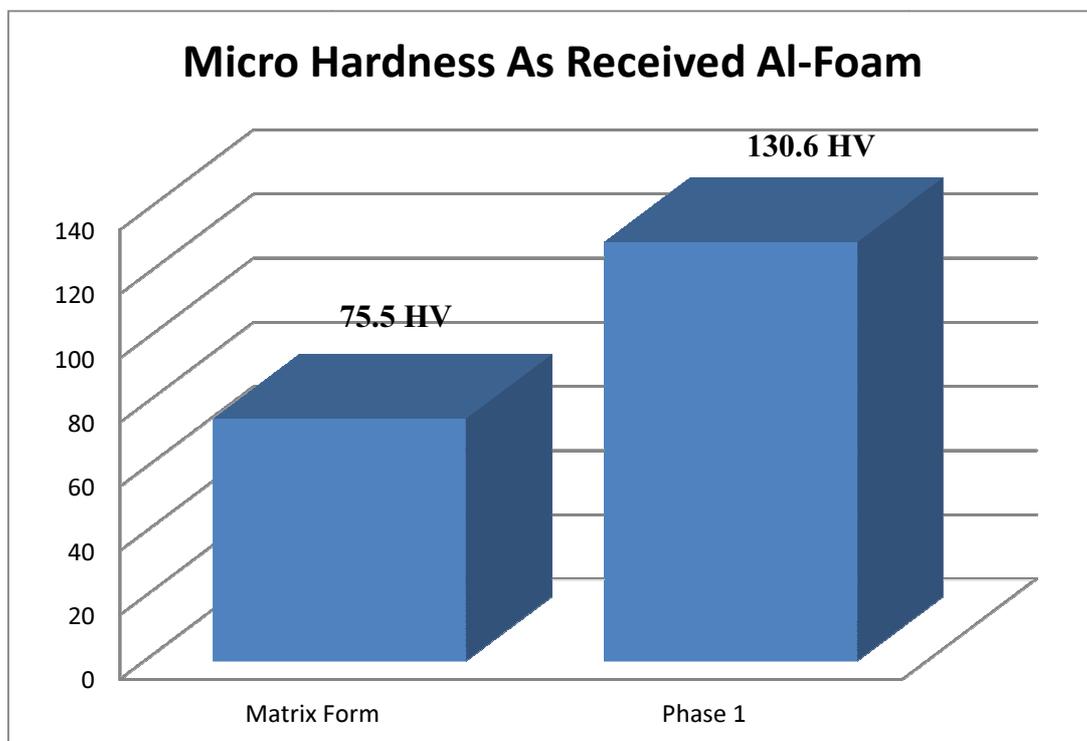


Figure 137: Micro hardness of as received Al-Foam

- **Brazed Heat Treated Aluminium Foam**

It was observed multiphase structure in brazed heat treated aluminium foam such as pure phase 1, the mixture of this phase with matrix and its matrix structure. They are shown in the following figure 138. The dark structure is the second phase.

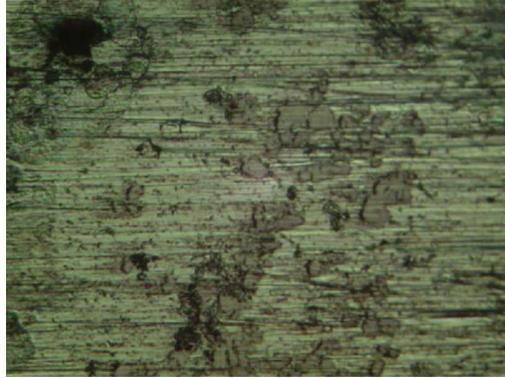


Figure 138: Multiphase structure of brazed heat treated Al-Foam

- **Matrix Form**



Figure 139: Matrix form of brazed heat treated Al-Foam

- **Pure Phase 1**



Figure 140: Phase 1 of brazed heat treated Al-Foam

- **The mixture of phase 1 with matrix**



Figure 141: Mixture form of brazed heat treated Al-Foam

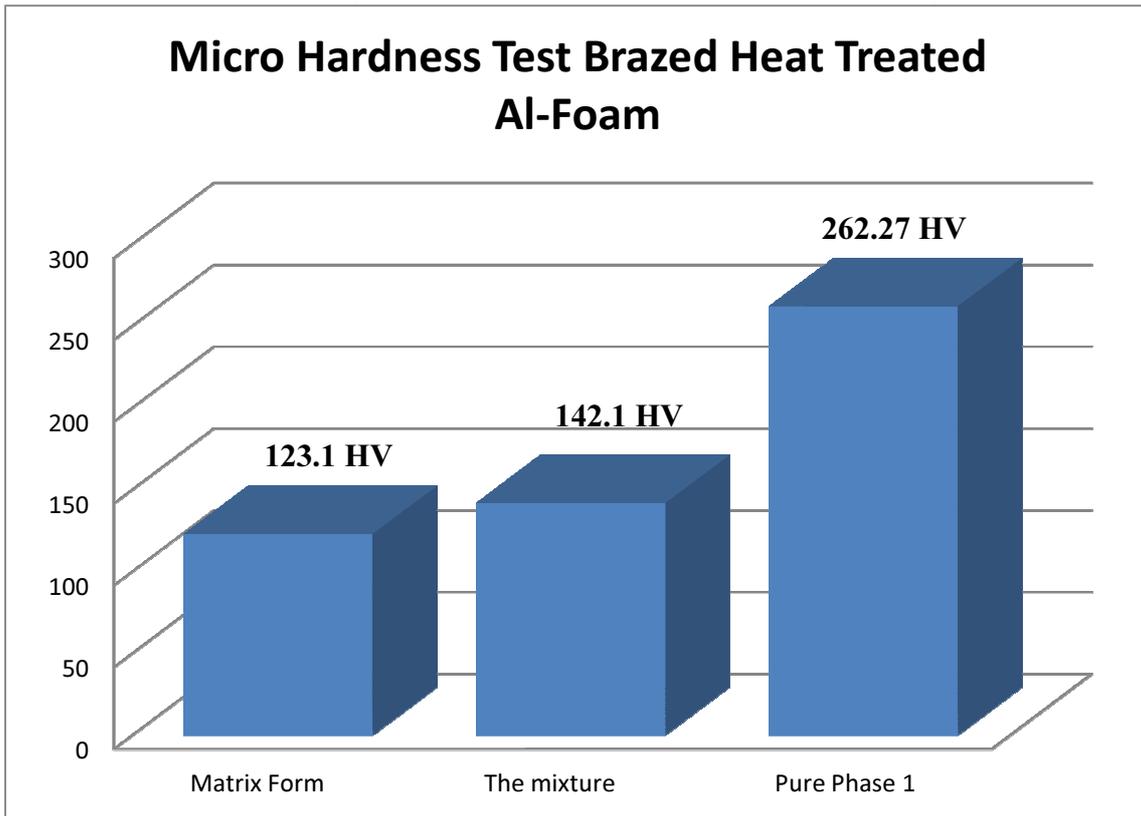


Figure 142: Micro hardness of brazed heat treated Al-Foam

The maximum average hardness value was obtained for pure phase 1 then the mixture of phase 1 with matrix form and lastly the matrix form, reported in the figure 142.

- **Solution Heat Treated Aluminium Foam**

It was also observed multiphase structure in solution heat treated aluminium foam such as pure phase 1, phase 2 and matrix structure. The dark colour areas are phase 1, inside the red circle meanwhile the light colour areas are phase 2, inside the black circle, are shown in the following figure 143.

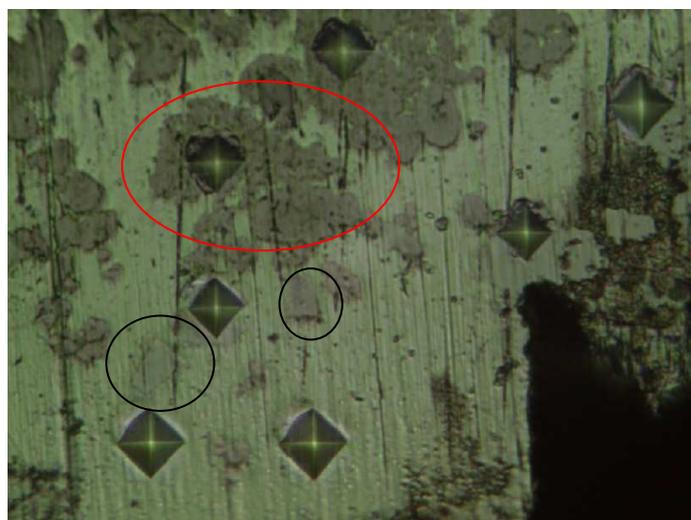


Figure 143: Multiphase structure of solution heat treated Al-Foam

- Pure Phase 1



Figure 144: Phase 1 of solution heat treated Al-Foam

- Pure Phase 2

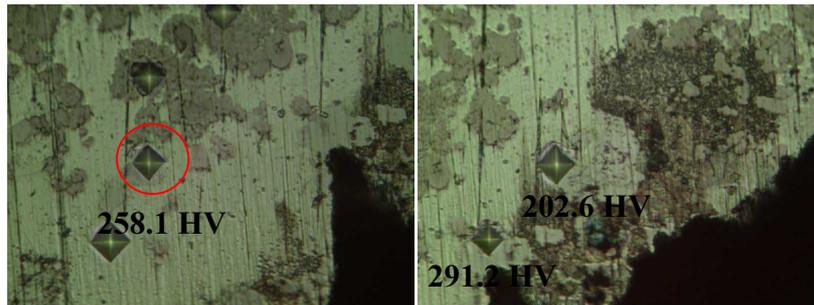


Figure 145: Phase 2 of solution heat treated Al-Foam

- Matrix Form



Figure 146: Matrix form of solution heat treated Al-Foam

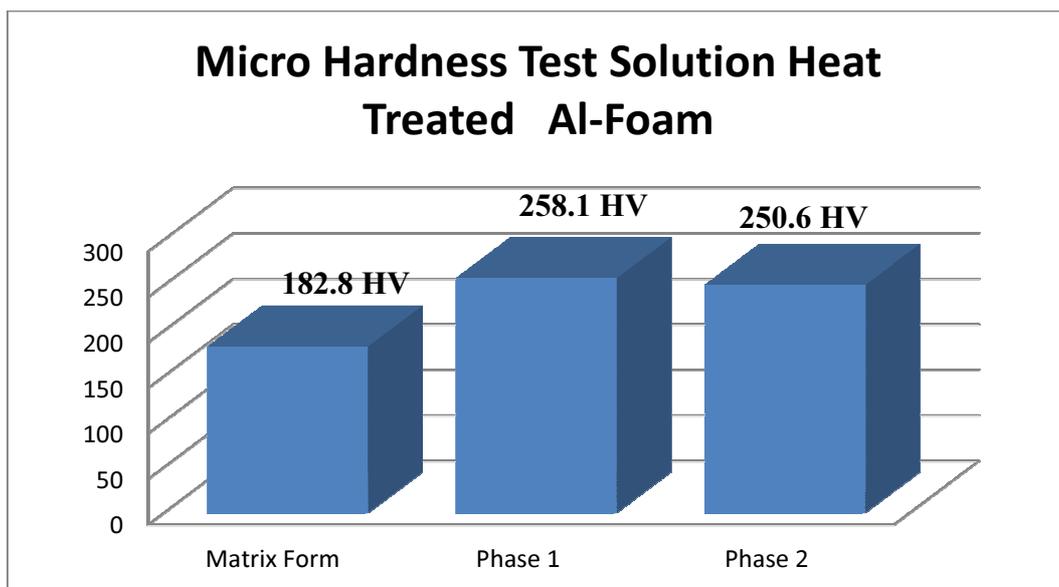


Figure 147: Micro hardness of solution heat treated Al-Foam

It is observed the increase of the hardness on the matrix structure of Al-foam when the solution heat treatment was implemented comparing with the brazed heat treated Al-foam.

# CONCLUSIONS

For brazed heat treated 1<sup>st</sup> batch Al-7046 alloys, it was seen the amount of Mg and Zn in the alloy composition became more appear. Also, it was observed the decrease on the strength around 28% and on micro hardness around 26%. In order to gain both strength and hardness, solution heat treatment was performed with water quench. From the results of the solution heat treatment, it was observed the amount of Zn and Mg in the alloy structure became less active. And with solution heat treatment, the increase on the strength was obtained around 10% of the brazing heat treated Al-7046's strength. Also, increase on the micro hardness around 28% of the hardness of brazing heat treated Al-7046. It was understood that solution heat treatment was a good application in order to gain strength and hardness after having decrease with brazing heat treatment.

As following to experiments in order to find the optimal parameters of furnace both for brazing treatments and solution heat treatments for producing AFS composites both brazing with pure Zn and Zn2%Al, when the parameters were decided, these new brazing and solution heat treatment parameters were applied on the new 2<sup>nd</sup> batch Al-7046 alloys in order to see the effects of new parameters in terms of strength and hardness on Al-7046 alloys.

From the results obtained for the 2<sup>nd</sup> batch Al-7046 alloys, when brazing heat treatment was conducted for the Al-7046 alloys, the decrease of strength was around 54% and on micro hardness around 34% when compared with as received samples. In order to gain both strength and hardness, solution heat treatment was conducted with water quench again. At this time, the increase of the strength was around 46% of the brazed heat treated 2<sup>nd</sup> batch Al-7046's strength. Also, increase on the micro hardness around 25% of the hardness of brazed heat treated 2<sup>nd</sup> batch Al-7046. The decrease of strength and hardness after brazing heat treatment were higher on 2<sup>nd</sup> batch than 1<sup>st</sup> batch due to brazing heat treatment temperature was higher for 2<sup>nd</sup> batch Al-7046 alloys. However, recovery amounts of strength and hardness after solution heat treatment were higher on 2<sup>nd</sup> batch Al-7046 alloys as well.

For AFS composite brazed with pure Zn, micro hardness test were applied one by one to the different regions in the joining area such as Al-plate, diffusion zone, Al-foam, joining seam. When the solution heat treatment was applied on the AFS composites brazed with pure Zn, it was observed the increase of hardness for all the regions in the joining area. The increase was 7% on the Al-plate, 56% on the diffusion zone, 93% on the Al-foam and 79% on the joining seam. However, it was seen the decrease on strength after solution heat treatment.

For AFS composite brazed with Zn2%Al, average strength of the specimens were increased by 48% comparing to the AFS composite brazed with pure Zn. However, after solution heat treatment, it was observed again the decrease of the strength on the specimens. Micro hardness test were applied one by one to the different regions again in the joining area such as Al-plate, diffusion zone and observed the increase of hardness for all the regions in the joining area. The increase was 16% on the Al-plate, 54% on the diffusion zone. It was also seen that the hardness values of Al-plate and diffusion zone were higher when AFS composite brazed with Zn2%Al rather than pure Zn.

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# ANNEX

## 1. Cutting Operations

Cutting operation was performed for the 3 different Al-7046 plates, as received one, brazed heat treated one and solution heat treated one with the dimension of 1mm to 1mm. 2 samples with the dimension of 1mm to 1mm were obtained from each groups. These, total 6 samples with the dimension 1mm to 1mm were cut as a first step for the analyses of electron microscope, micro hardness test, optical microscope, EDS and SEM.

Also, 3 samples from each group with the dimension of 60mm to 20mm were cut for the 3 point bending test. So, it was obtained that 3 samples 60mm to 20mm from as received Al-7046, 3 samples from brazed heat treated and last one 3 samples from solution heat treated. It is important to have at least 3 samples from each group in order to comparison in the group and having more data for analyzing. Also, it is essential to have precise dimension of 60mm to 20mm for bending as literature recommended it for conducting 3 point bending test.

All the cutting operations were performed by cutting machine which the name is ATA Brillant 220, see figure 148. The machine consists on control screen and set up screen, emergency stop buttons, blade height adjustment button, security door which is locked during the operations and below the security door, there are two control hoops for the adjustment of the table, which the part is assembled on the clamp mechanism, on the axis of x and y.

There are 2 different kinds of blades that ceramic one and diamond one were provided for the cutting operations in the laboratory. There are 2 diamond blades on the table, see the figure 148. Assembling of the blades into the cutting machine was done by wrench tools.



Figure 148: Ata Brillant 220 cutting machine

Aluminium plates were cut by a diamond blade with the 0.025mm/sec blade velocity. It is really important to obtain proper cutting speed otherwise it might have some problems on the blade, cutting machine or on the part which is being cut. During the operation, cooling of the blade and the part are also taking big importance and cooling is done by the two pipes into the rotation blade with the cooling liquid which is the mixture of distilled water with lubricant oil.

You can also select the type of the cutting operation on the control screen such as diagonal, vertical, both x-and y-axis, so on and the adjustment of the distance of the table which has to move on x-axis and blade height distance which how much distance blade has to go down in the part on the y-axis. Also, it is seen the water supply for cooling on the control panel with the figure worms-like black box. Always, it is important to be sure that water supply is on before running the machine.

As following the end of the cutting operation, I obtained total 9 samples with dimension 60mm to 20mm, 3 samples as received, 3 samples brazed heat treated and 3 samples solution heat treated. Now these samples are ready for the 3 point bending test. On the other hand, I obtained total 6 samples with the dimension 1mm to 1mm, 2 samples as received, 2 samples brazed heat treated and 2 samples solution heat treated. And these samples are ready for the epoxy resin and hardener coating process on the way of the preparation for optical microscope, micro hardness, SEM, EDS.

Briefly the steps of the operation on cutting machine are;

1. Assembly of the cutting blade into the machine and assembly of the sample on the clamping mechanism
2. Setup all the data and liquid supply check on the control screen
3. Alignment of the blade to the point which the cut conducts, it is made by the control hoops, moving the table on left to right and forward to backward. Arranging the liquid pipes that liquid should come to the blade
4. Last alignment check for the point then closing the security door of the cutting machine
5. Pressing the start button for the operation and alignment control during the operation out of the security door.
6. When the operation is over, pressing the stop button and opening the security door.
7. Removing the part from the clamping mechanism and removing the blade, cleaning inside the cutting machine and switch it off.

Cutting operation was done again with the same cutting machine, Ata Brillant 220, for as received Al-7046 plate and Al-foam. As received Al-7046 plate which has got 10cm to 15 cm dimension was cut to small pieces of 3cm to 2 cm dimension and Al-foam as well was cut to small pieces of 3cm to 2 cm dimension for brazing joining trails.

Pure Zn and Zn2%Al brazing joining component were cut by scissors due to being soft material. The same cutting parameters were conducted as before, 0.025 mm per second for Al-7046 plate and 0.035 mm per second for Al-foam.

After finding the optimum furnace parameters in order to produce AFS composite with furnace trials with these small dimension AFS composite, other cutting operations were obtained for real sized AFS composite for 3 point bending test. At that time, all the components of AFS composite were cut with the dimension 60mm to 20 mm with the same parameters.

## 2. Calibration and Cleaning of Furnace

Furnaces always should be clean and calibrated before any operations for that reason, the tube of the furnace was cleaned by metal stick which paper towels were rolled on it and alcohol was poured on the paper towels.

After cleaning operation for the tube of the furnace was done, when the paper towels were checked, it was seen black colour on it which might be some particles remaining from the last experiment or other factors. So, it is always essential to do cleaning operation before putting the samples inside the furnace tube.

If cleaning was not conducted, it might had failure of joining for AFS composite, over heat, degradation, oxidation or air vents on the surfaces during brazing joining treatment due to unknown remaining particles.

Another important issue is the calibration of the furnace that before having any experiment, it is essential to know the temperature difference between the real temperature inside the furnace tube and the one which was set on the screen of furnace.

For calibration of the furnace, which I used for brazing joining treatment was the name Carbolite Tersid, thermocouple was used, shown in figure 149, and the temperature on the thermocouple was the real temperature inside the furnace tube. It was needed to make a chart for heating, dwell and cooling to see the real temperature and its corresponding temperature on the furnace screen.



Figure 149: Thermocouple

The calibration process was implemented that way; Rod of the thermocouple was inserted inside the tube of the furnace and had connection to the thermocouple by light blue wire. Then all the machines were turned on.

Water supply was also turned on for avoiding over heating on the furnace exit pipe surface, shown with water hose in figure 150, the trial parameters for furnace were set on the furnace screen as following; temperature 750 °C, 20 minutes dwell time at maximum temperature, heating rate 16.6 °C per minute, cooling rate 10 °C per minute. Initial temperature inside the furnace was 22.5 °C. The temperature data were recorded with the temperature on the furnace screen and the real temperature inside the furnace tube on the thermocouple between 100 °C and 750 °C.

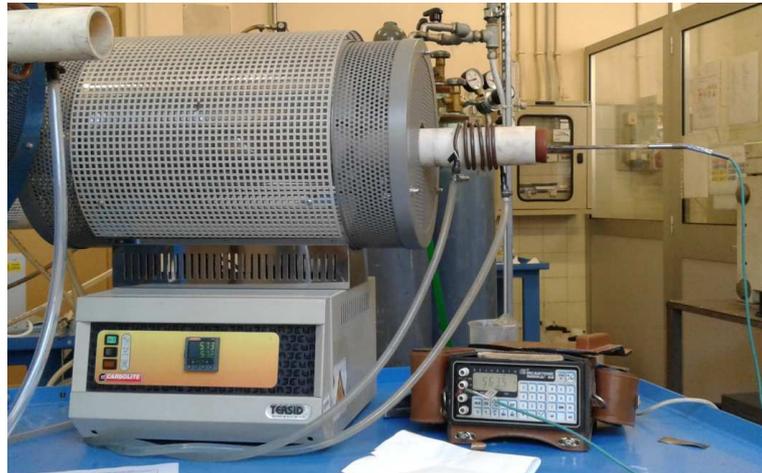


Figure 150: Calibration of Carbolite Tersid furnace

End of this process, I obtained all the temperature charts for both heating and cooling of real temperature inside the furnace and the temperature on the screen of the furnace. Then, it was seen the temperature difference between the one set on the screen and real temperature inside the furnace tube. After, these charts were used to find corresponding desired temperature for the furnace for all the experiments of producing AFS composite, see small section table 25.

Table 25: Small section from the calibration table

<b>Carbolite Tubular Furnace Calibration</b>			
<b>Carbolite °C</b>	<b>Thermocouple °C</b>	<b>Carbolite °C</b>	<b>Thermocouple °C</b>
100	45	540	436
110	50	550	447
120	56	560	457.5
130	60.5	570	469
140	66	580	479.5
150	71	590	490.5
160	77	600	501.5
170	83.5	610	512
180	89.5	620	523
190	96	630	534.5
200	103.5	640	545

### 3. Positioning of Sandwich Composites in the Furnace

When all the components were ready for the furnace brazing treatment, graphite paper was put under the Al-7046 sheet and graphite base structure is used in order to have flat platform before placing the AFS components inside the furnace. Also, another graphite paper was used on the top of the components. Then tungsten furnace bulks (balls) was put on the top of the components in order to give little pressure to downwards for joining between Al-foam and Al sheets when pure Zn or Zn2%Al were melted.

Consecutively for producing the AFS composite, from bottom to top; graphite bulk, graphite paper, Al plate, pure Zn or Zn2%Al, Al-foam, pure Zn or Zn2%Al, Al plate, graphite paper and tungsten bulks (balls) were used. Both graphite and tungsten materials have got really higher melting points than aluminium and zinc so they did not cause any problems.

For my trials to find optimum furnace parameters of brazing joining treatment for mass production, one side of AFS composites were produced in order not to waste materials. It is shown in the figure 151 below, consecutively, graphite bulk, graphite paper, Al plate, pure Zn or Zn2%Al, Al-foam and tungsten bulks (balls) were used.



Figure 151: Positioning of AFS components for trials

The heating resource of the furnace is located on the middle of the furnace tube. So, the AFS components should position on that area. After measuring the middle of the furnace tube about how much distance the components should move inside the tube, the AFS components were positioned by moving slowly the graphite bulk base structure with metal furnace stick, see figure 152. Now, components were ready for furnace treatment.

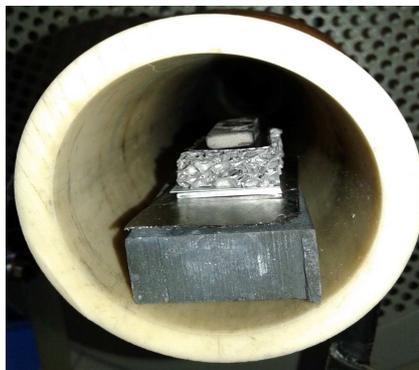


Figure 152: Positioned AFS components inside the furnace tube

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