PERFORMANCE EVALUATION OF COPPER BRAZING USING CU-NI-SN-P FILLER METAL

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FACULTY OF ENGINEERING
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ABSTRACT

Joining pure copper by brazing techniques using electric heated tube furnace has been done to brazed copper for heat exchanger application. Brazing temperatures specify within range of 660 °C to 700 °C using two different commercial alloy metals Cu-Sn-P base, VZ2200 and MBF2005. The filler metals were places between copper substrates layer. Heating processes was conducted in tube furnace at specific holding times of 5 to 15 minutes for each type of filler metal. Microstructure observation of the joints visibility was done using light optical microscope (OM) and field emission scanning electron microscopy (FESEM) and the element composition was analyzed by energy dispersive spectroscopy (EDS). Universal testing machine (UTM) was used for the inspection of mechanical property specifically for shear strength to measure the performance of the joints. Capture image under OM with the assist of computer software was conducted to measure the profile of intermetallic compound of the brazed layer. The brazed copper using filler metal of VZ2250 exhibited maximum shear strength 148 MPa when brazed at 680 °C with holding time of 15 minutes. While brazed pure copper using filler metal of MBF 2005 show its maximum shear strength 168 MPa when brazed at 680 °C with 10 minutes holding time.

Keywords:
Copper
Filler metal
Brazing temperature
Holding time
Microstructure

PENILAIAN PERKEMBANGAN PENDAKAP KUPRUM MENGGUNAKAN ISIAN METAL CU-NI-SN-P

ABSTRAK

Penyambungan kuprum tulen dilakukan dengan kaedah pendakap menggunakan relau pemanas elektrik untuk tujuan penggunaan komponen penukar haba bagi komponen sambungan kuprum.Suhu pendakap telah ditetapkan dalam lingkungan 660 °C hingga 700 °C menggunakan dua jenis isian metal berasaskan Cu-Sn-P yang tersedia dipasaran. Isian metal VZ2250 dan MBF2005 telah diletakan diantara permukaan kuprum yang hendak disambungkan.Proses pemanasan menggunakan relau tiub dibiarkan dengan masa penahanan yang spesifik antara 5 hingga 15 minit untuk kedua-dua jenis isian metal tersebut. Penelitian terhadap struktur mikro bagi penyambungan boleh dilihat menggunakan mikroskop cahaya optic dan mikroscop (OM), lapangan pancaran imbasan electron (FESEM) dan analisis pecahan elemen dengan menggunakan EDS.Mesin ujian universal (UTM) digunakan utk penelitian kekuatan ricih bagi perkembangan sifat mekanikal bagi kekuatan penyambungan kuprum. Imej yang diimbas oleh OM dengan bantuan perisian computer profil lingkungan antara metalik bagi lapisan pendakap penyambungan tersebut.Pendakap kuprum menggunakan isian metal VZ2250 mencatatkan kekuatan ricih tertinggi iaitu 148 MPa apabila suhu pendakap pada 680 °C dengan masa tahanan 15 minit, Manakala pendakap kuprum menggunakan isian metal MBF2005 mencatatkan kekuatan ricih tertinggi 168 MPa apabila suhu pemanasan pendakap pada 680 °C ketika masa tahanan 10 minit.

Keywords:
Kuprum
Isian metal
suhu pendakap
masa penahanan
Struktur mikro

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LIST OF SYMBOLS AND ABBREVIATIONS

°	:	Degree
µ	:	Micro
%	:	Percent
Ar	:	Argon
at	:	Atomic percentage
B	:	Boron
Be	:	Berylium
C	:	Carbon
C	:	Celcius
Ca	:	Calcium
cm	:	Centimeter
Cr	:	Chromium
Cu	:	Copper
EDS	:	Energy dispersive spectroscopy
FESEM	:	Field emission scanning electron microscopy
H	:	Hidrogen
K	:	Kelvin
K	:	Potassium
Li	:	Lithium
Mg	:	Magnesium
Min	:	Minutes
mm	:	Milimetre
MPa	:	Megapascal
Na	:	Sodium

Ni	:	Nickel
O	:	Oxygen
P	:	Phosporus
Pa	:	Pascal
RPM	:	Revolution per minute
SEM	:	Scanning electrons microscopy
Sn	:	Tin
T	:	Temperature
Ti	:	Titanium
UTM	:	Universal testing machine
W	:	Watt
wt	:	Weight percent
BFM	:	Brazing filler metal
IMC	:	Intermetallic compound

LIST OF APPENDICES

CHAPTER1: INTRODUCTION

1.1 Study Background

This study observe on joining performance of two metals. The selection type of joining method is brazing, a process of joining 2 or more metal using filler. Brazing is suitable method for joining metal due its convenience, cost effectiveness and high quality process. The selected material in this study is pure copper (99.99% wt). The samples of same pure copper brazed using two different type of filler. Commercial alloy filler used to join the two copper. The most important criteria of joining material investigation is their morphology. Brazing of the element of the iter first wall by rapidly quenched filler metals. Optimization of the composition of alloy filler metals based on the Cu-Sn-P system. Conducting metallographic investigations of the zone of brazing. This study will focus to determine the effect of heat treatment such as temperature and holding time on intermetallic compound and mechanical property i.e shear stress of the Cu-Cr-Zr (its composition). The temperature and holding time of annealing were chosen with consideration for brazing regimes of a particular product. Previous study discover temperature is raise adequate to melt the filler metal (Kalpakjian,2006). Related filler and it composition shown in Table 1.1.

Table 1.0: Filler metal composition

Composition (wt %) Composition Fillers				
Material	Nickel (Ni)	Tin (Sn)	Phosphorus (P)	Copper (Cu)
Filler VZ 2250	7	9	6	78
Filler MBF 2005	5.7	9.7	7.0	77.6

In this study the brazing factor i.e brazing temperature and holding time investigated to identify the performance of brazing on the two part joining copper surface using two different filler. The process of brazing temperature set at 660° C, 680°C and 700° respectively. The holding time for the process set at 5,10 and 15 minutes. In this study involve 9 samples for each filler, means in total 18 samples brazed in the tube furnace for each investigation i.e shear test, optical microscope (OM) analysis. The optimum sample select to be further investigated using field emission scanning electron microscope (FESEM) and energy dispersive spectroscopy (EDS).

1.2 Scope and limitation

The study involve new commercial alloy filler Cu-Ni-Sn-P used to joint pure copper (99.99% wt) with pure copper (99.99%wt).During experiment the temperature of the furnace increased until liquidus temperature of the filler metal is reached. Then the molten filler metal will flow into the gap two substrates by capillary action. Selected new foil type (40micron) of filler metal containing Cu–7.0Ni-9.3Sn–6.5P (VZ2250) and Cu–5.7Ni–9.7Sn–7.0P (MBF2005) is heated base on three level of brazing temperatures (660, 680,700 ° C) and treated by three differences holding time (5,10,15mins) were selected in order to investigate that quality and reliability of brazed joint.

The dry-argon purge into tube furnace during experiment to reduce the oxidation effect. The effect optimization of the composition used filler metals based on the Cu–Ni–Sn–P system will investigate. Comparative tests of two filler metals of brazing of pure measure by their mechanical properties i.e. shear test. The metallographic investigations of the zone of brazing joint will be observe using light Optical Microscope (OM), Field Emission Scanning Electron Microscope (FESEM) and Energy Dispersive X-ray Spectroscopy (EDS). This research will focus to determine the effect of difference brazing temperatures and holding times treatment on the mechanical properties of the Cu/Cu-Ni-Sn-P/Cu joint.

1.3 Objectives

Successful research aims clearly target to achieve. This research also aims clearly objective to be set as follow:

1. To evaluate the mechanical property (shear strength) with annealing treatment using various temperatures also need to relate with the effect from filler metal

2. To investigate microstructure analysis using light Optical Microscope (OM), FESEM/EDS and IMC apparatus.

1.4 Organization of report

There are 6 chapters in this report. Different topics delivered during study done. The topics listing of brief content chapters as follow.

Chapter 1

In this chapter the background of the study to gives as guideline and ideas to get the whole pictures of this study be done.

Chapter 2

Focus on literature review from previous related studies. Previous reports been screen and wrote in this chapter as guideline to run this study to get the aim objectices.

Chapter 3

This chapter stated the procedures of experiment been done in this study. Base from previous research methodology were apply to this chapter.

Chapter 4

In this chapter all the result from the experiment reported according to the limitation objective as set in chapter 1.The output from finding represent in the form of figures, tables and schematic graft.

Chapter 5

The result finding of research being discuses related to the outcome with the theory

Chapter 6

The last chapter that conclude the overall results and discussion that found in this study.

Also provide suggestion for future improvement for next investigation.

CHAPTER 2: **LITERATURE REVIEW**

2.1 Introduction of materials

Copper widely used as a conductor of heat and electricity, as a building material, and as a constituent of various metal alloys, such as sterling silver used in jewelry, cupronickel used to make marine hardware and coins, and constantan used in strain gauges and thermocouples for temperature measurement. Copper is soft, malleable and one of the most ductile metals, thus copper can be shaped in to thin sheet, drown into wires and made into pipes. Strength and hardness are appreciably increased by cold-working because of the formation of elongated crystals of the same face-centered cubic structure that is present in the softer annealed copper. Common gases, such as oxygen, nitrogen, carbon dioxide, and sulfur dioxide are soluble in molten copper and greatly affect the mechanical and electrical properties of the solidified metal.

The unique performances of copper includes heat conductivity, higher electric conductivity and corrosion resistance makes copper widely applied in engineering structure (Liu et all. 2008).

The second half of last century, comprehensive studies of the coloration between mechanical properties and microstructure have been performed (Murphy 1981).

General fact copper are commonly alloyed with another metals to make it more reliable according to the needs. Tin, zinc or gold often used during alloying copper to become new metal entity such as bronze (alloyed copper with tin) and brass (copper with zinc).Brass copper produces for better corrosion resistance than pure copper (Earth science for society 2011). Copper alloy stronger and more ductile as temperature decrease.

2.2 Brazing

There are several method of joining two or more copper. Copper brazing normally used for heat exchanger, EGR cooler and dissimilar metal joining. Brazing is one of the joining process using filler metal placed in between the metal surface to be joined by melting and flowing a filler metal into the joint. Brazing involve heating temperature and time for the purpose of melt the filler metal. The melting temperature for filler metal typically above 450°C and below the melting point of base metal to be joined. The filler metal having a lower melting point than the joining metal. Copper alloy filler usually used to joint pure copper to other metal. Flux is often used during brazing. It is a liquid that promotes wetting, which lets the filler flow over the metal parts to be joined. It also cleans the parts of oxides so that the filler bonds more tightly to the metal parts. In addition, fluxes are used in welding to clean the metal surfaces. Soldering temperatures and joining strength are lower than brazing (Kalpakjan 2006). The filler metals are mainly alloy of silver, aluminum, nickel, cobalt or copper (Sulzer Metco 2011). The important factor to achieve good joint ability of brazing are influenced by the filler properties, wetting and spreading by molten filler metal and the interfacial reaction between the molten filler and interfacial reaction between base material and filler material (American Welding Society Handbook AWS 2007).

2.2.1 Cycle of Heating

There are varies of heating cycle depend on the study case. The study of heating cycle for 304 stainless steel plate-fin braze with BNi2 filler metal conducted by Jiang, Giong and Tu (2011) use seven steps. Beginning with the step by pumping vacuum of the furnace to 0.001 Pa, then the stacked structure of plate–fin is heated up to 850° in 50 minutes. The third step the temperature is held at 30 minutes to reduce the gradient temperature for the whole joints. After that, step four set the heating is resumed until brazing temperature reach 1050°C during 30 minutes, and it will lead the best capillary flowage of nickel-base filler metal.

The fifth step brazing time controlled in 25 minutes to control the thickness of the base metal and the complexity of the joints. The carbide of the austenitic stainless steel achieved during this cycle by preferable solid solution treatment. Sixth step of vacuum self-cooling while the temperature reduces from1050 to 620°C within 40 minutes. The final step while lowest temperature reached 620°C, immediately the quick cooling is performed using dry nitrogen by filling into the furnace. The wind cooling system also take places at same time starting up within 50 minutes. The whole body temperature set about 40°C, plate-fin brazing structure removed from the furnace then lets cool in the air.

The same procedure conducted in another research by Miab and Hadian (2014) shows similar techniques. The case of braze cubic boron nitride (cBN) /steel (CK45) joint with Cusil-ABA uses using active brazing filler alloy. A constant brazing temperatures of 920° was set-up during brazing process in a tube furnace surrounded high purity argon atmosphere and the brazing time range between 5 and 15 minutes.

2.2.2 The temperature effect

The most important parameter that effect the brazed joint performance is brazing temperature. The range of temperature depending on type of metal be joined and the filler are used. Difference value of temperature give different value of effect such as microstructure and strength of brazing. Temperature has big influence to change the microstructure and mechanical properties of brazing metal. The case brazing of sapphire with Inconel 600 by variation of 830-900°C using thin sapphire and thin Inconel 600. The fail brazing result occurred at low brazing temperature 830° due to high residual stress. Meanwhile successful joining result archived at high temperature 900°C as the specimen could preserve (T.Zaharinie et al, 2014).The higher the temperature gives the higher strength of joining (J Liu et al, 2013). High brazing temperatures inhibit the joining to the base metal and the fluidity of brazing filler metals diminishes with increasing Cu content.

The studies on graphite and copper using Ni-Cr-P-Cu filler alloy also supported the effect of brazing temperature. The number and size of Cu-based solid solution increase while the brazing thickness layer reduces when the brazing temperature increased. The interface of graphite and filler alloy reaction layer increases effect the thickness of brazing result. Result shown the higher brazing temperature faster reaction speed between the elements (Zhang et al.2014).

High brazing temperatures inhibit the joining to the base metal, and fluidity of brazing filler diminishes with increasing Cu content (Tatsuya et al 2014). Apart from that, the joint strength will be affected by spreading area and wetting angle of the copper base alloy filler.

Surface roughness of the base metal consider as another important role in braze joint strength and spread ability of filler. Shabtay and co-worker have reported that copper and its alloys can withstand high-temperature brazing process without substantial loss in strength (Y.L Shabtay et al 2004). A study reported that the successful brazing and good wetting achieved by the least void by using and intermediate surface roughness (average Ra value) and the Ra value around 0.20 µm found to the most appropriate for brazing of copper conducted at the specified process parameters (T. Zaharinie et al, 2015). Similar finding have also been reported by other research which indicate the the molten filler metal is able to wet a rough surface well rather than a fine surface (Y. Li et al, 2012).

2.2.3 Holding time effect

A research done by Miab and Hadian (2014) to study brazing time effect on the mechanical and microstructure properties changes. Cubic boron nitride metal are chosen to joints using active brazing alloy, Cusi-ABA. The result of the study shown that the increment of brazing time significant gave changing of microstructure brazed layer.

The increment of brazing time will affect the braze layer microstructure. The changed can be identified by observation on dendritic shape on the dark phase translocate and concentrated across the interfaces. The reaction of Ti and cBN caused the phenomenon during brazing process. Ti is an important element as activator to react with cBN to form wettable portion. This Ti activate element used as interfacial reactions during brazing process. Increase of dwell time will affect the change of Ti to diffuse toward cBN/Cusil-ABA interface to produce Ti-rich phase along the interphase.

Copper brazing holding time allow sufficient period for molten filler metal to interact with the base metal. The wetting behavior of the filler can investigate using light optical microscopic (OM) that shows an effective spreading flow of the filler metal over the base metal. The strong braze result achieved when the filler metal has diffused into the base metal through the grain boundaries. This strong braze joint having a high shear strength.

2.3 Mechanical Properties of Joints

Pure copper joints brazed with amorphous Cu68.5Ni5.7Sn9.3P6.5 filler metal using vacuum furnace investigated by Jing Zhang show that the tensile strength affected by increased and decreased time and brazing temperature. The maximum value of tensile strength 135MPa of brazed copper was found when brazing performed at 680 °C for 20 min (J. Zhang et al 2013).

Cu-P filler metal are used due to their low melting point, superior wettability, perfect self- fluxing and low cost. Content of P 5wt % or more filler alloy used in brazing copper, a brittle intermetallic compound (IMC), Cu3P tend to form at room temperature.

A new brazing cycle design for brazing copper been develop by T Zaharini and team which involve the best process parameter 650 °C and holding time 5 minutes. The shear test result for this parameter gave the highest value of 190 MPa in the braze joint (T Zaharini et al, 201).

Very difficult processing but discover new kinds of new Cu-P filler metal been manufactured using rapid solidification (W. Liu et all 1990). The brazing joint using Cu-Ni-Sn-P amorphous filler metal been investigated by influence of brazing temperature and time to the phase constitution and tensile strength. The effect of brazing time on joint strength at temperature 680 ° C increase up to 20 min to 30 min and give the maximum value of tensile strength 135.19 MPa for 20 min. Short brazing time (5min) leads to insufficient atomic diffusion between the melting metal and copper substrate just give the value of tensile strength lower as 80.5MPa (J Zhan et al, 2013).

2.4 Phase diagram

Phase diagram is basic foundation material investigation guide in performs basic material research include solidification, joining, phase transformation, crystal growth, solid state reaction and etc. Phase diagram plays as a road map for process optimization and materials design as it is the starting point in the manipulation of process variables in order to achieve the desired microstructure (Austin Chang, 2004).

The element relationship show in phase diagram includes temperature, the composition and the phase present under equilibrium condition for the specific alloy. Known as an equilibrium or constitutional diagram, phase diagram refers to it equilibrium phase for the metal. It stable condition present equilibrium as defines the static or constant state of a system that maintain over an indefinite period of time. For constitutional indicate the link between the composition, structure and physical make-up of the alloy (Kalpakjian, 2006).

Distinguish of phase diagram by it characteristic of a metal to change the phase of solid solution with the other materials. The characteristic listed in 3 conditions

Condition 1: two metal are completely soluble in each other in all proportions in the solid state.

Condition 2: two metals are completely insoluble in each other in the solid state

Condition 3: two metals are partially soluble in each other in the solid state

The microstructure will be produce when alloys cooled under equilibrium conditions. The microstructure indicate by the alloy phase diagram.

2.5 Intermetallic compound

Intermetallic compounds are defined as solid phases involving two or more metallic or semi metallic elements with an ordered structure and often a well-defined and fixed stoichiometry (C.C Barlow 2001). Intermetallic compounds have a high melting point and are generally brittle at ambient temperature. They often offer a compromise between ceramic and metallic properties when hardness or resistance to high temperatures is important with sacrificing some toughness and process abilities (K. Suganuma 2013).Intermetallic is an electrochemical bonding between the brazed and the copper surface.

It takes place during reflow when the Tin in the filler reacts with the Cu substrate or layer. In Sn-rich solders on a Cu substrate, Cu_6Sn_5(η) or Cu_3Sn (ε) intermetallic layers are formed at the solder/substrate interface.

2.6 Optimum process parameter of brazing copper

Set of pure copper metal selection for purpose of braze using filler metal was conducted by Tuan Zaharinie and team test brazing process using vacuum furnace at temperature range 650-750 °C. The holding time was set at 5-15 minutes. During the study they concentrate on surface roughness investigation as main effect to relate with the strength of joint.

The best brazing parameter has found at highest shear strength of 190MPa in the copper braze joint. The successful brazing and good wetting can be achieved by the least voids by using an intermediate surface roughness and the Ra value around 0.20 µm was found to be the most appropriate for brazing of copper conducted at the specified process parameters (Zaharinie et all. 2015).

CHAPTER 3: **METHODOLOGY**

3.1 Preparation of Sample

The brazing procedure begin with the selection of the metal to be joined. This study used two pieces pure copper (99.9 % W) during brazing process. Selected upper using copper rod to be cut into small pieces sized 8mm diameter x 5mm thickness and for the lower copper substrates the size is cut into 15mm x 15mm x 3mm.The sample assembly dimension show in Figure 3.1. The copper surface clean up and grind using 1500 grade sandpaper to become mirror image. The grind sample then clean and dry up in a safe dry place to avoid oxidation and unwanted impurities on the material.

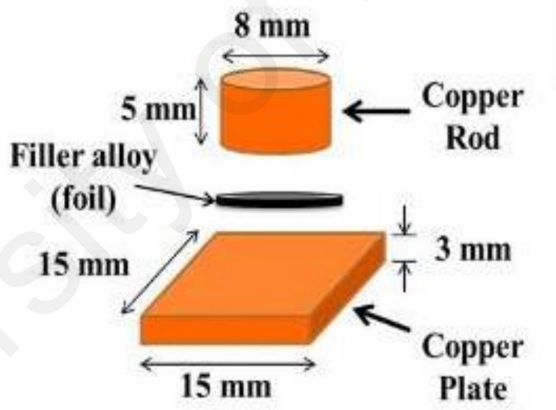


Figure 3.1: Dimension of sample assembly preparation

Carefully preparation of surface to be joint will get optimum strength of brazed copper. Thus in order to have good sample the surface must be checked before applying filler metal for brazing purpose.

Two copper alloy filler as brazing metal filler (BFM) being use in the study. Selection of commercial filler metal of VZ2250 (filler 1) and MBF 2005(filler 2) foil type thickness

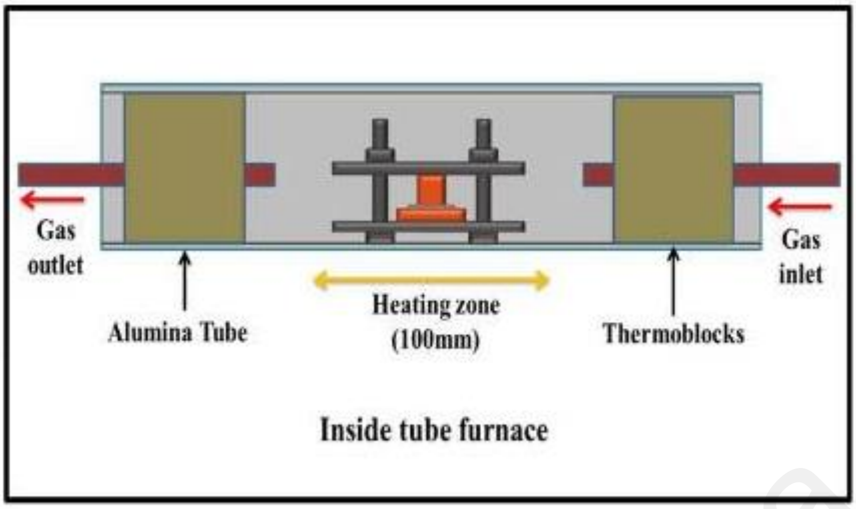
40 µm expected as suitable filler metal for copper joint purpose of heat exchanger application. The filler metal liquidus at 630 °C and solidus at 600°C. The foil filler cut to size 10mm diameter X 40µm of thickness. Double ply of filler prepared to be placed at the surface of joining pure copper. It means that the thickness of filler metal become 80 µm

3.1.1 Cleaning Solution Preparation

The copper surface must be clean up by using cleaning solution. The cleanliness of this metal surface to avoid any unwanted impurity or dirt and to get better join. Cleaning solution prepared using sulphuric acid and distils water. The portion of 5 ml sulphuric acid and 95ml distils water mix in a container. The solution mixture ready to clean the sample. The copper soak in the solution container for two minutes. Then the copper remove from the solution using tweezer to get it dry.

3.1.2 Sample Preparation

Cleaned and dried material is a must procedure to join metal. Two copper sample arrange layer by layer to form of sandwich type. Square size pure copper placed at the bottom part, then foil copper alloy filler and cutting rod copper on the top. Complete sandwich assembly are clamped by a jig before brazing procedure as shown in Figure 3.2. This assembly are ready to be braze in a vacuum tube furnace.



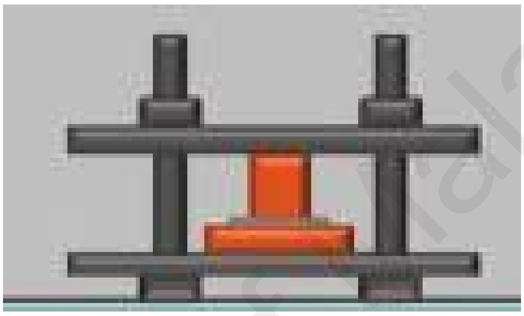


Figure 3.2 Sample clamp using jig inside tube furnace

3.2 Brazing Procedure

The join copper braze in a furnace, KYK tube uses argon gas as protective agent atmosphere gas. Clamped sandwich inserted into the furnace tube. The sample are pushed and placed to the center of tube by using a cylindrical rod. Furnace tube tightly closed. Sample place in furnace arranging and programmed according to the study requirement as stated in Table 3.2. This study set the brazing temperature at 660°C,680°C and 700°C and holding time at 5, 10 and 15 minute. The same brazing parameter used for brazing copper using 2 different brazing filler metal, VZ2250 and MBF 2005 commercial copper alloy filler.

Table 3.1: Programmed brazing temperature and holding time

Sample	Brazing temperature (°C)	Holding time (min)
1	660	5
2	680	5
3	700	5
4	660	10
5	680	10
6	700	10
7	660	15
8	680	15
9	700	15

3.3 Heating process

Heating for brazing purpose involve two important parameter, temperature and time. The programmed procedure set up according to the requirement of the join profile segment illustrated in Figure 3.3. There are 4 segments for brazing process of copper using alloy filler in a furnace. Segment 1 as example the temperature set to 30°C let the furnace initial temperature rise up to 30 °C before heating process begin. Temperature increasing at segment 2 up to require setting temperature (e.g. 650°C, 680°C,700°C) and control by certain temperature rate (°C/min). For illustrated sample takes 5°C/min it means 5 °C increase in a minute. Different set of sample the heating temperature will have different heating time. The target of heating temperature for sample reached, the burning in furnace will stop for a specific period of time. This is represent by segment 3 known as holding time. Each sample set the holding time (5, 10, 15 min) as stated in Table 3.2. After holding time completed the temperature will decrease for cooling purpose. Segment 4 show the cooling stage by controlling the temperature decreasing rate as example 5°C/min, means 5°C decrease in a minute. The complete process of brazing copper went through up 4 stages involve pre-heating, heating, holding and cooling steps.

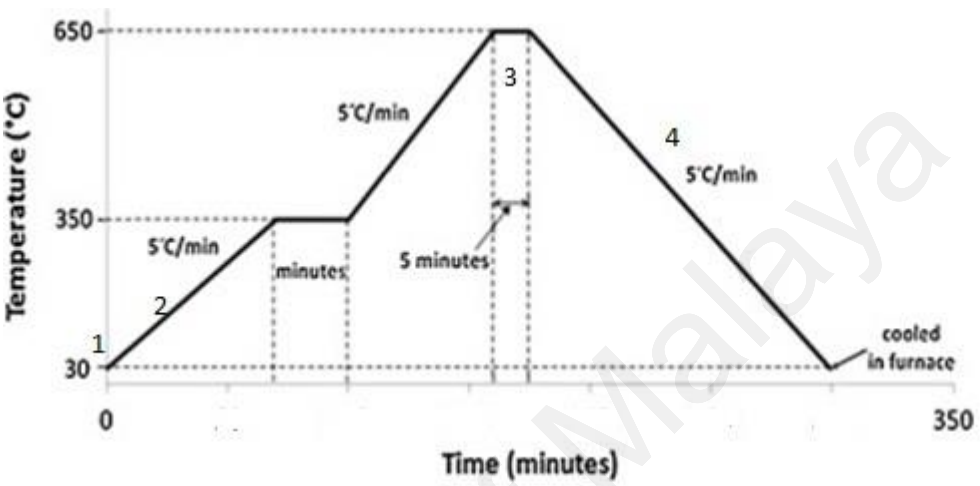


Figure 3.3: Profile segment sample illustrate copper brazing at 650 °C temperature and 5minutes holding time

To perform shear strength for braze copper, samples directly test after complete brazing process from furnace without any mounting procedure. Shear test using universal testing machine with 3 samples for each test. In total 27 samples of filler VZ2250 and 27 samples of filler MBF2005.

3.4 Mounting procedure

The brazed sample cool by air-cool then to be mount. Mounting the sample to get easier handling grip during preparation and investigation purposes. The sample is cut, grind and polish for the investigation. Mounted sample prepare by few apparatus consist of resin, plastic container, hardener and wooden stick.

All the apparatus must be in cleanliness condition. Dry and clean plastics container being use to mix the mounting ingredients before pour into brazed sample in mold. The portion of 8 spoons of resin and 2 spoons of hardener mix together in plastic container. The mixture being stir to blend and make sure no present of bubble. Blending mixture then pour into mold with the brazed sample inside. Make sure the mixture are fully cover the sample in the mold. This procedure finish by given twelve hours to harden itself.

3.5 Cutting process

Harden mounting sample removes from the mold to cut for microstructural observation purpose. A cross section cut begin with marking point then clamp with a jig for easy cut process. A jig tighten using screws by mounting sample align to cutting blade. Linear Precision Machine being use to cut the clamped sample. The cutting machine use diamond blade for cutting process.

Machine speed and feed rate must be consider during this process. The copper mounted cutting for this sample is set the blade rotation speed 280 rpm with feeding rate about 2mm per min.

3.6 Grinding and polishing

Excellent surface layer is a requirement for best observation of microstructure. Best preparation to get best result the sample begin with grind and polish. To grind copper sample selection of high grade of sand paper is choose to avoid surface scratch. Five grades of sand paper being use to grind the cutting sample. Begin about three minutes grinds by using 600 grade follow by 800 grade for same rate. After 600 and 800 grade completed the repeat process by using 1500 and 2500 then 4000 grade. While using high grade, 2500 and 4000 sandpaper grinding time increased to 20 minutes. High grade of sandpaper and longer time of grinding process could get better surface good condition better as mirror image surface.

Grinding machine shown in Figure 3.4, set at low speed about 250-300 rpm for low grade sandpaper (600, 800, and 1500). High speed of 400 rpm set for high grade sandpaper (2500, 4000). High speed will assist to get faster good surface condition. To avoid heat occur from friction on the surface, tab water is use to cool the heat between sample and sandpaper.

Figure 3.4: Grinding and polishing machine

The same grinding machine as shown in Figure 3.4 is use for polishing process .Sandpaper is not required for the process. Polishing the sample uses ultra –gum cloth with additional of micro polish with metal solution. Two micro polish solution being use 3µ and 1µ diamond suspension. Polish 3µ of diamond suspension for 5 minutes follow by 1µ suspension for the same 10 minutes polishing duration. The complete polish sample observe under optical microscope (OM) to make sure no scratch surface, void, dirt's and unwanted impurities before etching process.

3.7 Etching Sample

After OM observation shown that complete polish sample in optimum condition etching is ready for next process. In order to get best quality surface condition etching purpose are to clean and remove unwanted dirt or impurities on the surface by using chemical solution. The etching solution prepare using three component, sulphurid acid, distils water and iron chloride. Solution mixture consist of 100 ml distils water mix with 50ml sulphuric acid and 5g of iron chloride. The mixture stir adequately before use to etch the sample. The prepared portion of solution can use for etch 3 samples. Polished braze copper sample are soaked for 5 second sink in the solution. Sample remove from the solution clean by distils water and dry up using hair drier fan. Etching sample kept in dry container with silica gel as shown in Fig 3.5 to avoid contaminant and oxide surface. The dry and clean sample can to be observe under OM and FESEM.

Figure 3.5: Container for sample

3.8 Testing Apparatus

Mechanical performance testing of shear strength are using universal testing machine (INSTRON) as shown in Figure 3.6. The machine set up with a low cross-head speed of 0.5 mm/min (Zaharinie et all 2015). Brazed sample being test after completely remove from furnace. The assembly of copper run the test without mounting procedure.

Figure 3.6: INSTRON universal testing machine

Light Optical Microscope (OM) been used for microstructure observation as refer to Figure 3.7. The sample grinded and polished then etched before perform OM test. OM link with computer software for image processing and analysis. Result can obtained from OM are images and sizes of profile.

Figure 3.7: Light Optical Microscope (OM)

Same sample used to performs microstructure investigation using field emission scanning electron microscope (FESEM). Surface copper brazed observation for the effect of brazing temperature and brazing time and its composition by energy dispersive spectroscopy (EDS) using the same table top machine. FESEM machine as shown in Figure 3.8.

Figure 3.8: Field emission scanning electron microscope (FESEM)

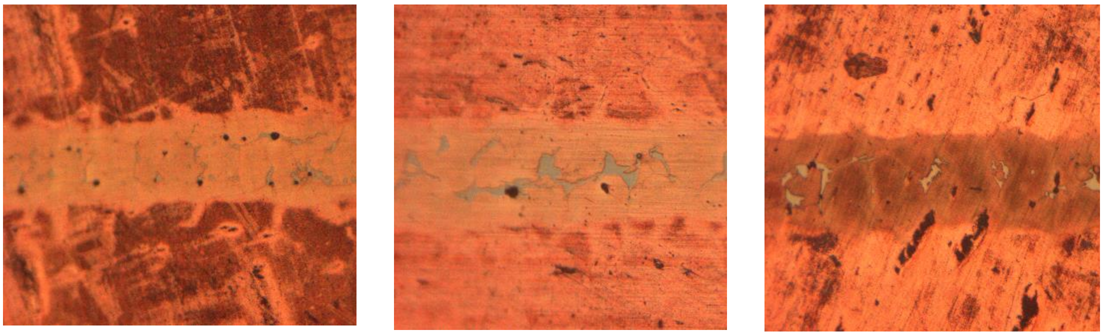
CHAPTER 4: **RESULT**

4.1 Light optical microscopy observation

Microstructural observation was performed with a light optical microscopy (OM) after the sample been grinded and polished. Before observation under OM to get it better image view etching was done to all samples. Each sample show different image of microstructure. The various pattern of image presented according to each brazing parameters such as temperature and time. The image of observation captured and presence in this chapter to identify the contras in each sample by measurement technique. The comparison method between each sample used to identify the effect of brazing parameter to the brazed copper.

4.1.1 Microstructure comparison within the same holding time

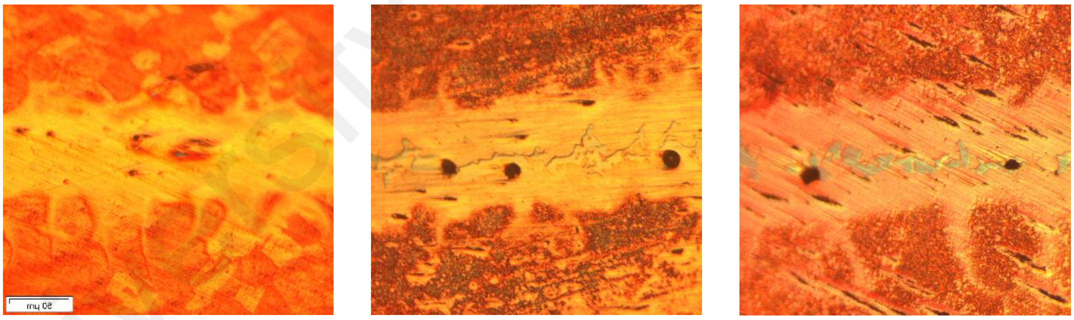
Brazing holding time for this experiment was set up to 5, 10 and 15 minutes for selected 2 type of BFM filler metal (filler metal 1 : VZ2250 and filler metal 2: MBF2005). The microstructure of brazing area for each filler also show different pattern related to alloy filler composition. The capture from OM camera can clearly show in Figure 4.1 for filler 1 VZ2250 for brazing holding time at 5 minutes.



a) 660°C b) 680°C c) 700°C

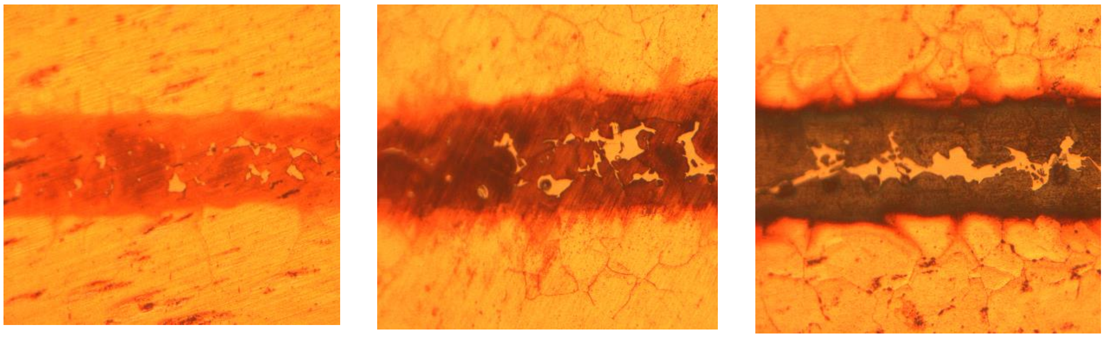
Figure 4.1: Brazing copper using filler metal of VZ 2250 for holding time 5 minutes

Time holding at 10 minutes for filler 1 VZ2250 show in Figure 4.2. Brazing temperature 700 °C clearly show that more grey area at brazing layer as compare to 680 °C and 660 °C.



a) 660 °C b) 680 °C c) 700 °C

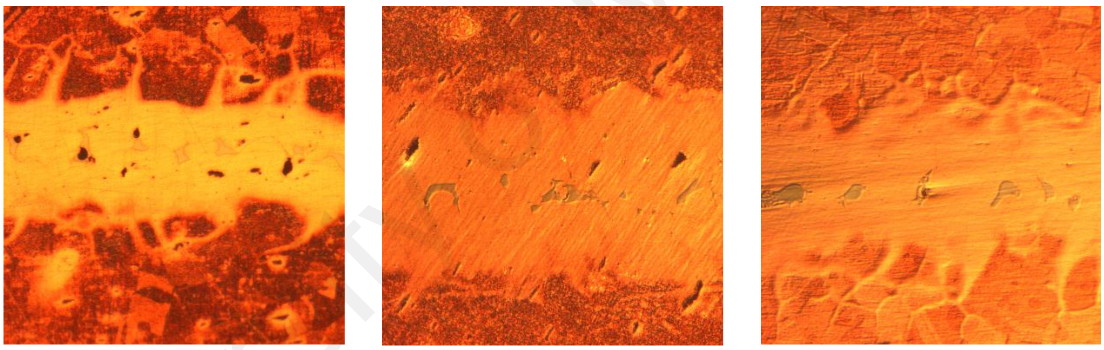
Figure 4.2: Brazing copper using filler metal of VZ 2250 for holding time 10 minutes



a) 660 °C b) 680 °C c) 700 °C

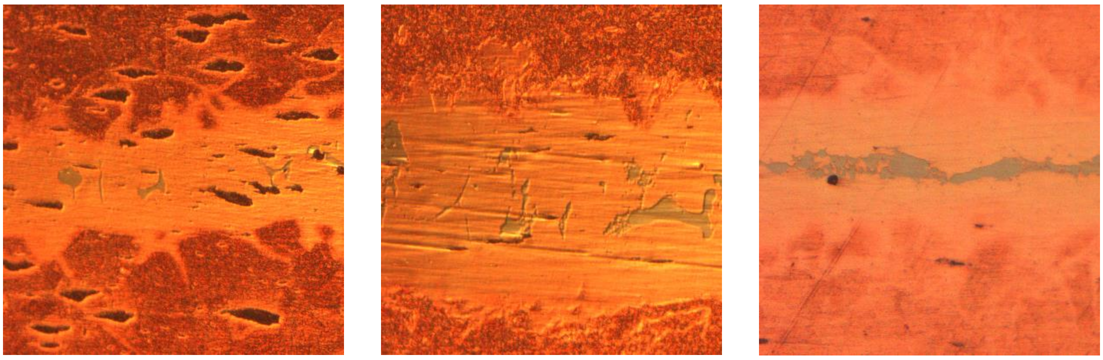
Figure 4.3: Brazing copper using filler metal of VZ 2250 for holding time 15 minutes

Optical observation result for brazing filler metal (BFM) of MBF 2005 copper alloy filler



a) 660 °C b) 680 °C c) 700 °C

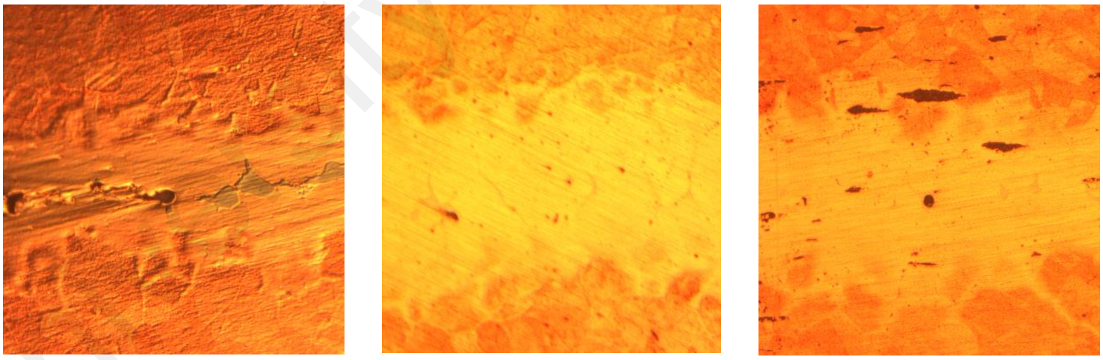
Figure 4.4: Brazing copper using filler metal of MBF2005 for holding time 5 minutes



a) 660 °C b) 680 °C c) 700 °C

Figure 4.5: Brazing copper using filler metal of MBF2005 for holding time 10 minutes

Brazing filler metal of MBF 2005 used at holding time 15 minutes show in Figure 4.6. At temperature 660 °C looks more continuous grey as compare to temperature 680 °C and 700 °C

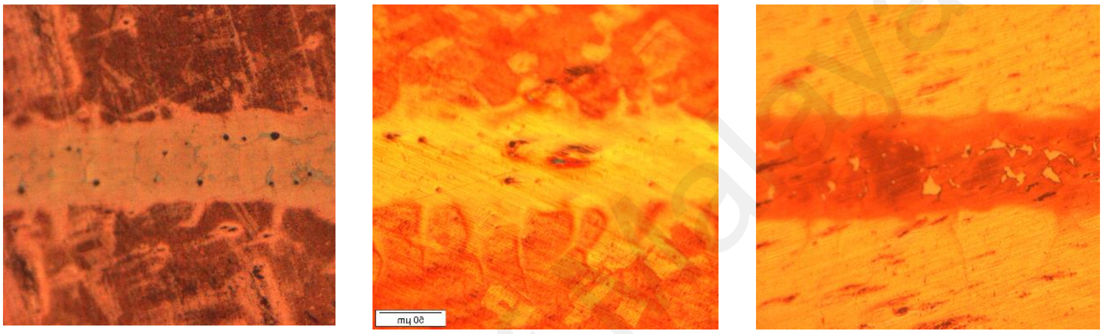


a) 660 °C b) 680 °C c) 700 °C

Figure 4.6: Brazing copper using filler MBF2005 for holding time 15 minutes

4.1.2 Microstructure comparison with the same temperature

Observation under OM can compare within similar temperature and different holding time. Brazing temperature was set up to 660 °C, 680 °C and 700 °C respectively. Microstructure of brazing copper show different pattern according to brazing temperature even the same period of time.

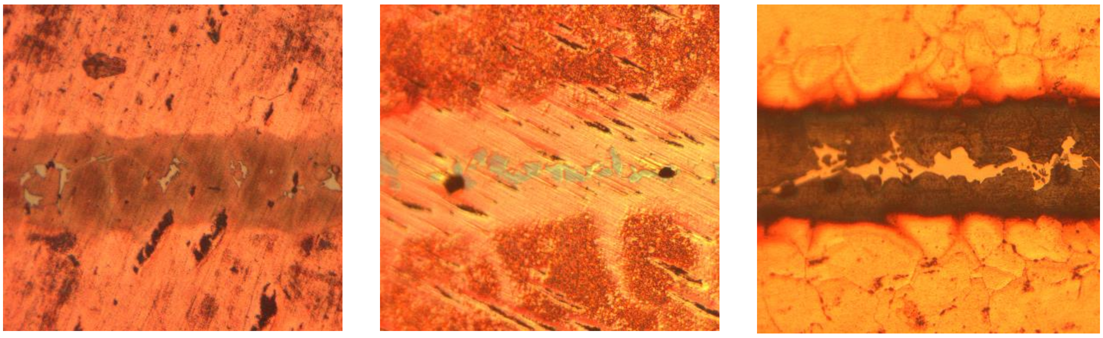


a) 5 min b) 10 min c) 15 min

Figure 4.7: Brazing copper using filler metal of VZ 2250 for brazing temperature 660 °C

a) 5 min b) 10 min c) 15 min

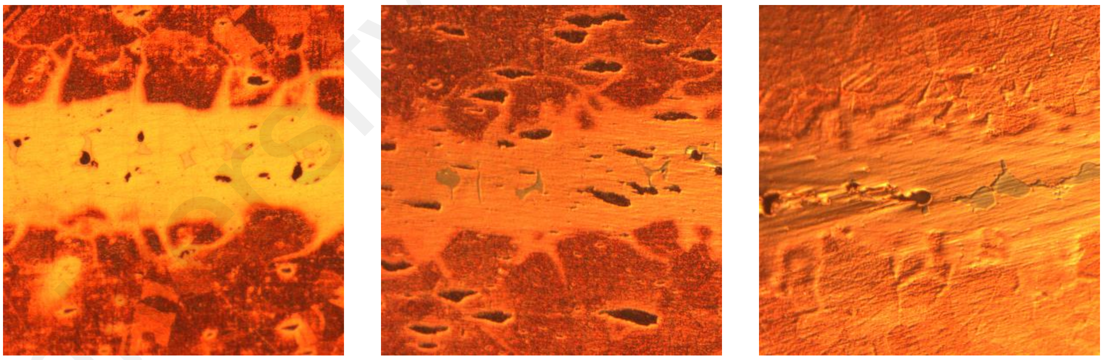
Figure 4.8: Brazing copper using filler metal of VZ 2250 for brazing temperature 680 °C



a) 5 min b) 10 min c) 15 min

Figure 4.9: Brazing copper using filler metal of VZ 2250 for brazing temperature 700 °C

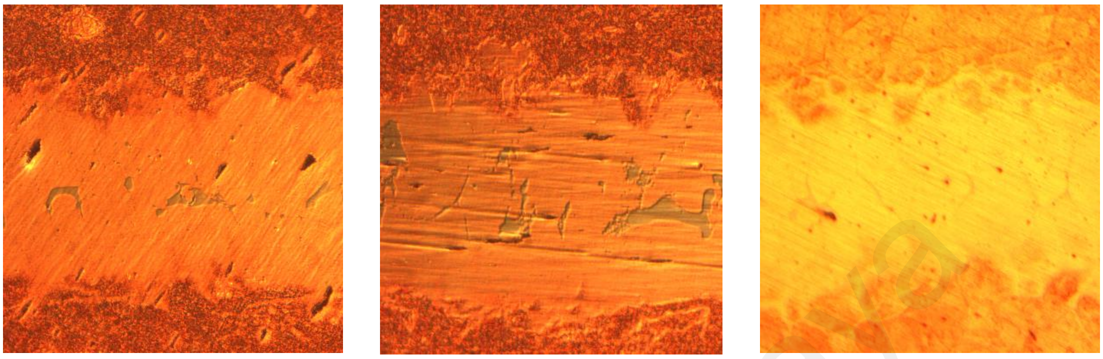
Optical observation result for brazing filler metal (BFM) of MBF 2005 copper alloy filler of temperature 660 °, 680 °C and 700 °C respective with indicated holding time 5,10 and 15 mins.



a) 5 min b) 10 min c) 15 min

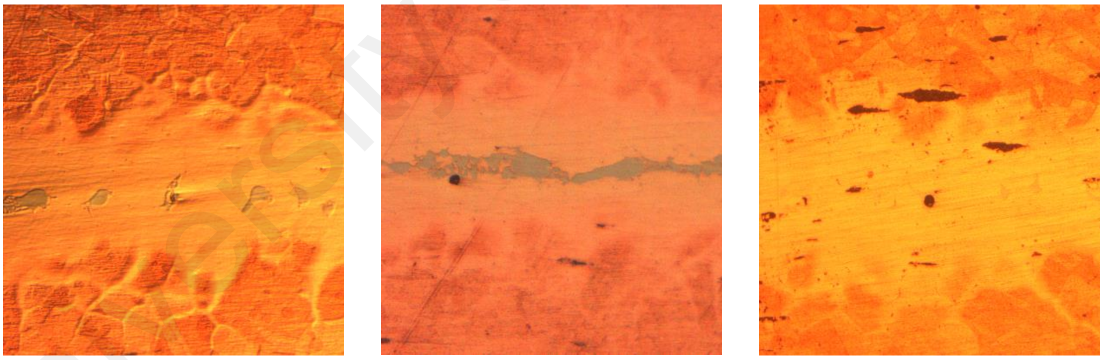
Figure 4.10: Brazing copper using filler MBF2005 for brazing temperature 660°C

Figure 4.11 and 4.12 show the different images of microstructure brazed at temperature 680 °C and 700 °C respective to the holding time of 5 ,10 and 15 minutes. The microstructure of joints brazed at different holding time for specific temperature shows the change of brazing layer thickness with the grey phase condition. At this temperature a large amount of continuous grey phase (Cu_3P) and show less scattered dark grey phase ($(CuNi)_2P$) former in between the copper subrates.



a) 5 min b) 10 min c) 15 min

Figure 4.11: Brazing copper using filler MBF2005 for brazing temperature 680°C



a) 5 min b) 10 min c) 15 min

Figure 4.12: Brazing copper using filler MBF2005 for brazing temperature 700 °C

4.2 Shear strength result

Mechanical property investigation for the brazed sample using INSTRON universal testing machine (UTM). Three sample been test for each brazing parameter set (temp °C/time min). The test run at 0.5 mm/min and the reading was recorded. Best two value from each set sample present in the report selected base on the best standard deviation value. Shear strength test result for BFM filler VZ2250 show in the Table 4.1

Table 4.1: The shear strength result for MBF filler VZ2250

Sample °C/min	Shear strength S1 (MPa)	Shear strength S2 (MPa)	Stdev	Shear strength Average (MPa)
660°C/5min	138.66	139.2	0.38	138.93
680°C /5min	146.86	144.16	1.91	145.51
700°C /5min	138.35	132.24	4.32	135.30
660°C /10min	137.68	133.39	3.03	135.54
680°C /10min	113.32	106.95	4.50	110.14
700°C /10min	110.52	110.16	0.25	110.34
660°C /15min	144.04	146.87	2.00	145.46
680°C /15min	145.07	150.51	3.85	147.79
700°C /15min	140.73	144.4	2.60	142.57

From the Table 4.1 the optimum value for shear strength available when the sample was brazed at 680°C at holding time 15 minutes. The highest shear strength for this brazing parameters is 147.79 MPa. Base on optimum shear strength values the brazed sample with same parameter 680 °C/15 min selected for further investigation using FESEM and EDX.

The following Table 4.2 show the shear strength test result for BFM filler MBF2005. The same procedure of testing sample using INSTRON universal testing machine at rate of 0.50 mm/min.

Table 4.2: Shear strength of braze copper using MBF filler MBF2005

Sample °C/min	**Shear strength S1 (MPa)**	**Shear strength S2 (MPa)**	**Stdev**	**Shear strength Average (MPa)**
660°C/5min	158.26	154.68	2.53	156.47
680°C /5min	157.33	146.94	7.35	152.14
700°C /5min	159.91	152.23	5.43	156.07
660°C /10min	122.09	132.51	7.37	127.30
680°C /10min	169.79	165.32	3.16	167.56
700°C /10min	149.1	148.05	0.74	148.58
660°C /15min	153.11	175.47	15.81	164.29
680°C /15min	142.99	154.36	8.04	148.68
700°C /15min	142.92	162.04	13.52	152.48

From the result refer to the table the highest value of shear strength show at the brazing temperature 680 °C and holding time 10 minutes. The shear strength value at this parameter is 167.56 MPa.

4.3 Inter metallic compound (IMC) profile

Microstructure investigation concentrate on brazing filler metal (BFM) layer. The layer within subtract copper lower and upper being investigated to profile the intermetallic compound (IMC) profiling. The layer that contact within copper and filler being measure and recorded. Figure 4.13 show the schematic brazed copper of IMC profile been measured. Brazing within 2 surface of copper (brazing layer) image capture under OM and measure using computer software. The measure layer marked as I (diffusion layer) II (brazing filler layer).

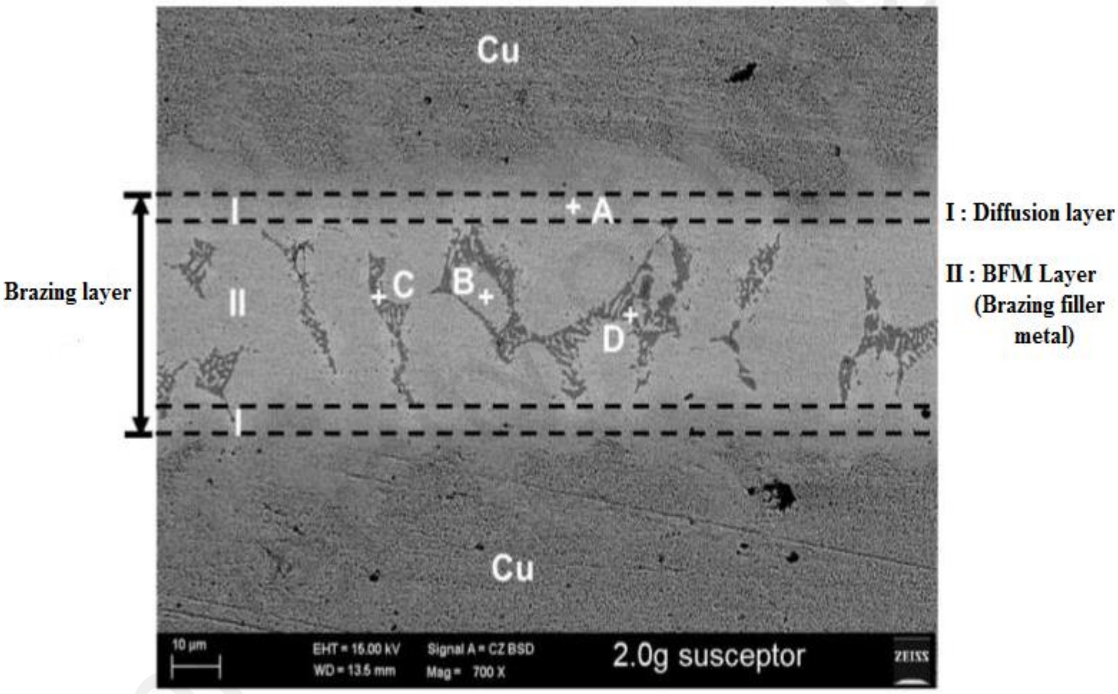


Figure 4.13: Schematic measurement of joint copper sample for IMC profile layer

Intermetallic compound (IMC) profile measurement brazing copper using brazing alloy filler VZ2250 layer result. The result of profiling measurement show in Table 4.3.

Table 4.3: measurement of brazing profile thickness of VZ2250 filler metal

Sample (°C/min)	Brazing layer (μm)	BFM layer (μm)	Diffusion layer (μm)
660/5	64.72	36.39	18.52
680/5	84.10	36.39	22.88
700/5	71.68	37.26	17.39
660/10	67.99	27.08	21.15
680/10	78.90	31.83	23.98
700/10	89.56	37.26	25.28
660/15	78.01	33.58	19.61
680/15	79.75	39.00	24.19
700/15	90.21	32.90	24.41

The profile thickness measurement of brazed copper using brazing filler metal of MBF2005 layer result. The result of thickness profiling measurement show in Table 4.4.

Table 4.4: measurement of brazing profile thickness for MBF2005 filler metal

Sample (°C/min)	Brazing layer (µm)	BFM layer (µm)	Diffusion layer (µm)
660/5	77.13	33.78	28.99
680/5	132.90	46.61	38.49
700/5	88.24	31.87	27.02
660/10	86.87	41.40	17.44
680/10	145.65	81.04	39.35
700/10	91.73	28.32	26.58
660/15	74.54	23.38	20.72
680/15	109.37	41.63	35.74
700/15	95.21	30.51	23.53

The table of thickness profile for both two filler metal of VZ2250 and MBF 2005 used to plot bar chart to discuss the research output in next Chapter 5.

CHAPTER 5: **DISCUSSION**

Result from the experiment discussed in this chapter. Discussion of research output present in this section assisted with graphics of figures, graph and bar charts.

5.1 Effect of brazing parameters to shear strength

As brief in previous chapter, brazing parameters will affect the properties of copper joints. This section focus on the affect brazing temperature and holding time to the mechanical property i.e shear strength. The data analyzed and plot by line graph to see the change and to discuss the effect of these two brazing parameter to shear strength. From shear test result it show the effect of brazing temperature within specific holding time.

5.1.1 Effect of brazing temperature

Brazing temperature plays the main factor of brazing parameter to the mechanical property (shear strength). From the experiment the output from the test carry out with the graph to see the different and changes of brazing temperature. Figure 5.1 show the plot of brazing temperature versus shear strength for brazed copper using metal brazing filler metal of VZ2250. From the result of the test found that the highest value of shear strength occur at brazing temperature 680°C at holding time 15 minutes of joint copper using brazing filler metal of VZ 2250. The highest strength recorded at this brazing parameter is 148 MPa.

The effect of this parameter caused joint crystallization process will increasingly with brazing temperature and directly and certainly degrade the mechanical properties of the joints (J. Zhang et all)

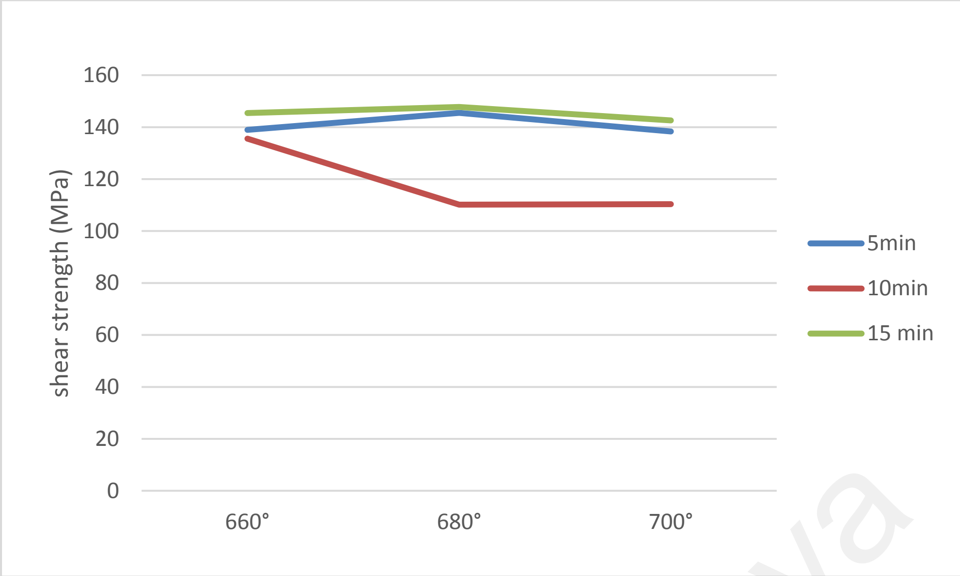


Figure 5.1: Result of shear strength test brazed copper at brazing temperatures ranges using filler metal of VZ 2250

Brazing copper using of MBF 2005 filler metal show the different pattern effect of brazing temperature to shear strength. Brazing sample in furnace temperature 680 °C at holding time 10 minutes get the highest value of shear strength. During brazing temperature 660 °C and holding time 5 minute appear the lowest shear strength and brazed at 700 °C show the average value for all holding time. The highest value of shear strength, 168 MPa held at 10 minute.

Figure 5.2 show the effect factor brazing temperature to mechanical property, shear strength for brazed copper using filler metal of MBF 2005.

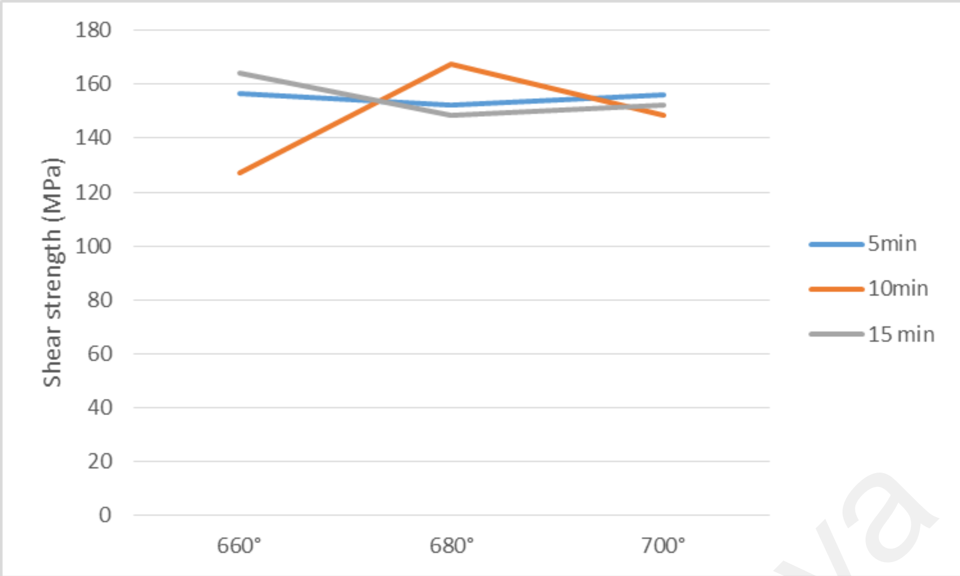


Figure 5.2: Result of shear strength test brazed copper at brazing temperature 660°C, 680°C and 700°C using filler metal of MBF 2005

5.1.2 Holding time effect

Holding time exerts liter influence compare to brazing temperature parameter. The sample test begin with 5 minute holding time show the shear value nearly same with all the brazing temperatures 660°C, 680°C and 700°C. However the second test attempt at holding time 10 minutes show little difference shear strength range about 15%. Holding time for brazing copper at 15 minute as can see from Figure 5.3 show that it appear the closer value of shear strength for all brazing temperatures parameter. From this investigation can see the prolonging of brazing time, especially 15 minutes for both alloy filler the thickness of the reaction layer similarly increase. Figure 5.3 and 5.4 show the evident of increasing holding time will increase value of the strength.

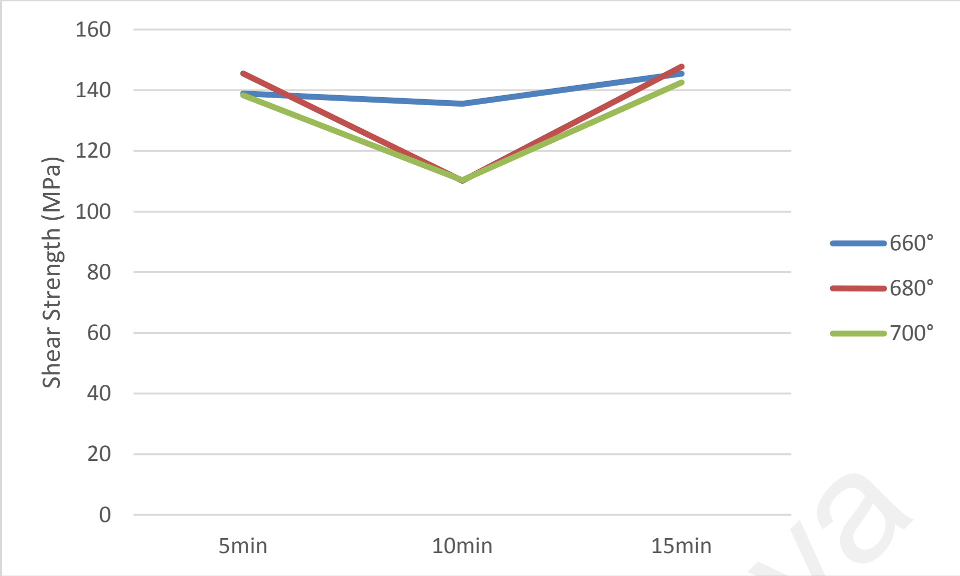


Figure 5.3: Effect of holding time to shear strength brazing copper using filler alloy VZ 2250

The brazed copper strength using filler alloy VZ 2250 archived the highest value of shear strength when the holding time set at 15 minutes. This is most suitable holding time for brazing copper compare other 5 min and 10 minutes. Shear strength show the lowest value when the holding time at 10 minutes due to the softening effect during brazing process. The highest strength brazed copper when holding time 15 minute brazing in furnace at temperature 680 °C is 148 MPa.

Brazed copper using brazing alloy filler MBF 2005 give the highest value of shear strength at 10 minute holding time. Figure 5.4 show the comparison effect of holding time for each brazing temperature 660 °C, 680 °C and 700 °C. From the graph clearly can see the highest shear strength value 168 MPa when brazing temperature 680 °C at holding time 10 minutes.

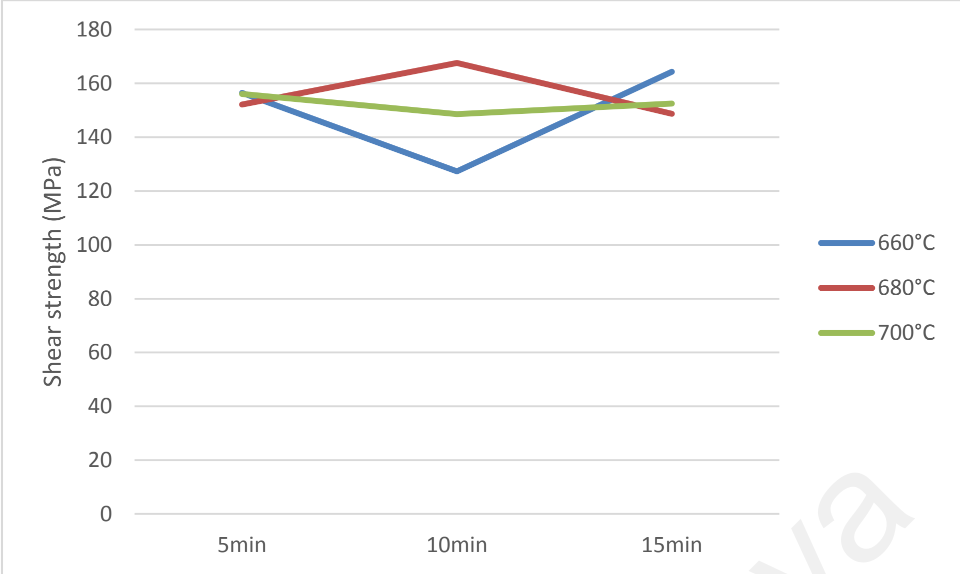


Figure 5.4: Effect of holding time to shear strength brazing copper using filler metal of MBF 2005

5.2 Intermetallic compound thickness profile region

Brazing within 2 surface of copper (brazing layer) image capture under OM and measure using computer software. The measurement layer list as brazing layer and diffusion layer for purpose of analysis. From result in chapter 4 the brazed copper investigation for brazing thickness profile plotted in the bar chart. Figure 5.5 show the brazing layer effect by brazing parameter (holding time and temperature) for filler metal of VZ2250. Brazing thickness show the highest value at 15 minute holding time. The thinnest layer occur when holding time at 5 minutes. Means for short period of holding time (5 minutes) leads to insufficient atomic diffusion in between the melting filler metal and the copper substrate. For the prolonging holding time it show the thickness of this layer increase. However further prolonging the holding time reduce the bonding strength because of grain growth.

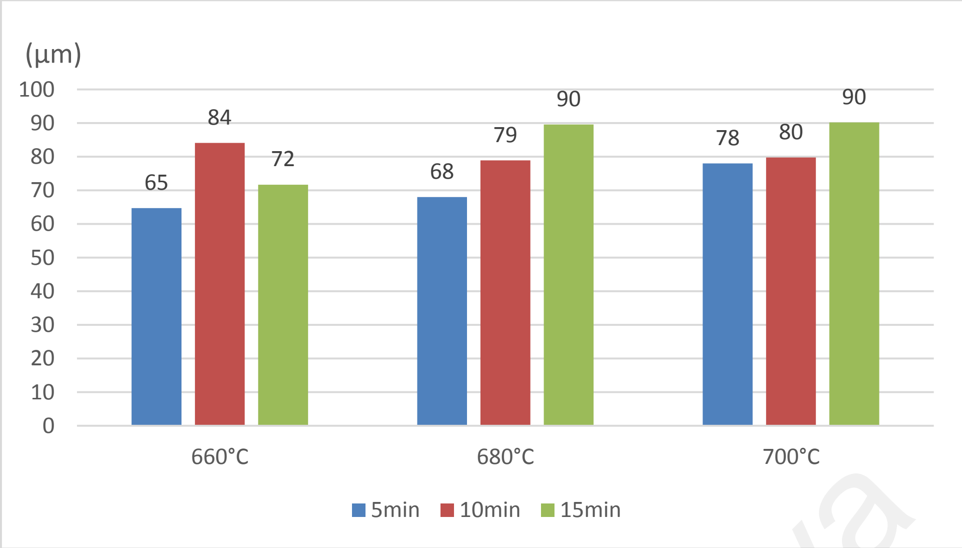


Figure 5.5: IMC profile brazing layer effect of brazing temperature using filler metal of VZ2250

Diffusion layer of brazed copper for filler metal of VZ 2250 recorded thickness range 27 – 44 µm. Prolonging times at 15 minutes show decrease of the thickness with while increasing of brazing temperature. It can be evidence that the thickness of residual filler metal layer decrease with increasing brazing holding time. By statement show that dwelling time does not affect to the formation of the irregular interface. Figure 5.6 show the diffusion layer thickness effect by brazing parameter of brazed copper using of VZ2250 filler metal.

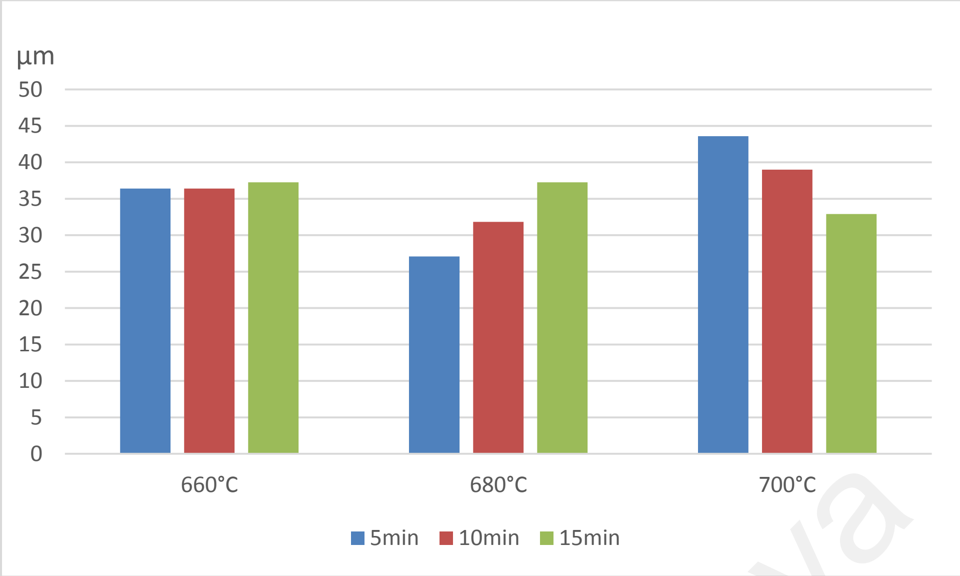


Figure 5.6: IMC profile diffusion layer effect of brazing temperature using filler metal of VZ 2250

The brazed copper using of MBF 2005 filler metal show brazing thickness highest value when brazing temperature 680 °C. The holding time at 5, 10 15 minutes show range brazing thickness of 109- 143 µm. By increasing the brazing temperatures can see that the brazing thickness decrease for all parameter of holding time. Figure 5.7 show the comparison of brazing thickness brazed at indicates temperature and holding time. From the chart it found that 10 minutes is suitable time for sufficient atomic diffusion of melting filler react with copper substrate at brazing temperature 680 °C. This ideal brazing parameter give the strongest bonding of joint for the brazed copper using the filler metal of MBF 2005.

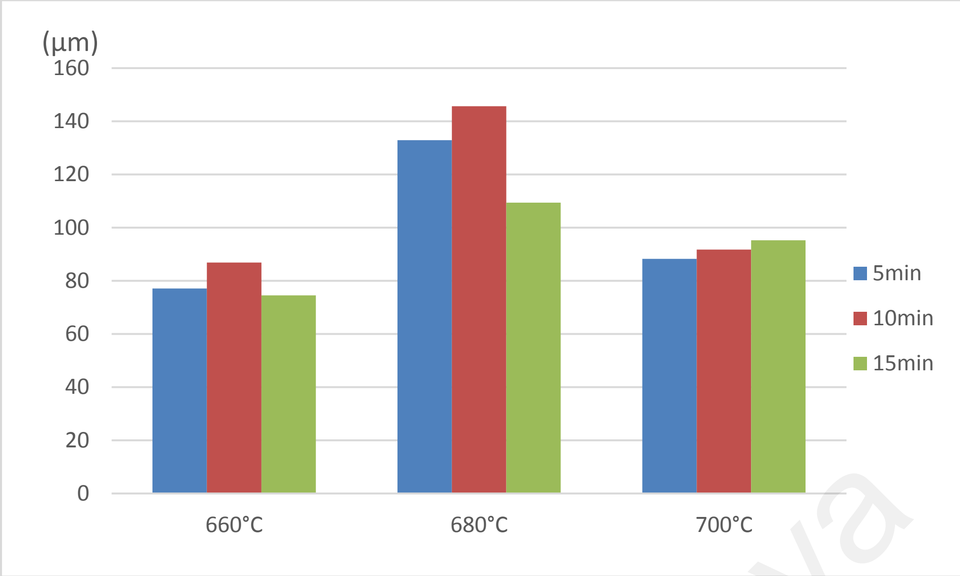


Figure 5.7:Brazing layer thickness for filler metal of MBF 2005

Diffusion layer for brazed copper using filler metal of MBF2005 show the quite similar pattern when brazing temperature 680 °C at all holding time. It decrease with the increase of the temperature.

From Figure 5.7 observation can conclude too short or too long holding time could not assist strong joint whereas a medium brazing time is capable to make a joint with higher strength.

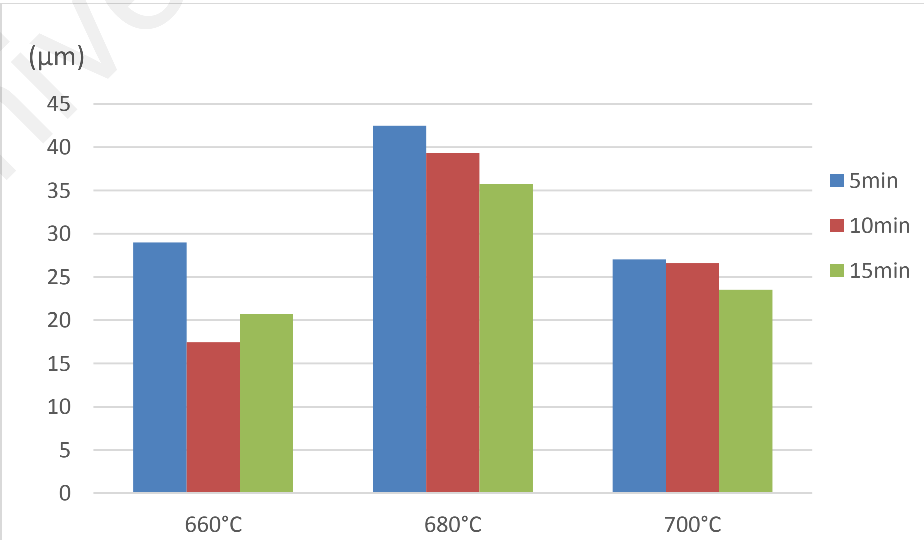


Figure 5.8: Diffusion layer thickness for filler metal of MBF 2005

5.3 EDS analysis

Microstructure investigation of brazed copper also observe under field emission scanning electron microscopy (FESEM) and EDS analysis being discus in this section. The sample tested select from optimum value of joint strength for both filler metals of VZ2250 and MBF 2005. Brazing copper using of VZ 2250 filler metal only the sample with brazing temperature 680 °C and holding time 15 minute been tested. For another filler metal of MBF 2005 joints the optimum sample select is 680 °C and holding time 10 minutes. Figure 5.9 and 5.10 show the optimum sample image and it element composition weight percent (%wt) at specific point marked as A, B and C using filler metal of VZ2250 (Figure 5.9) and MBF2005 (Figure 5.10)

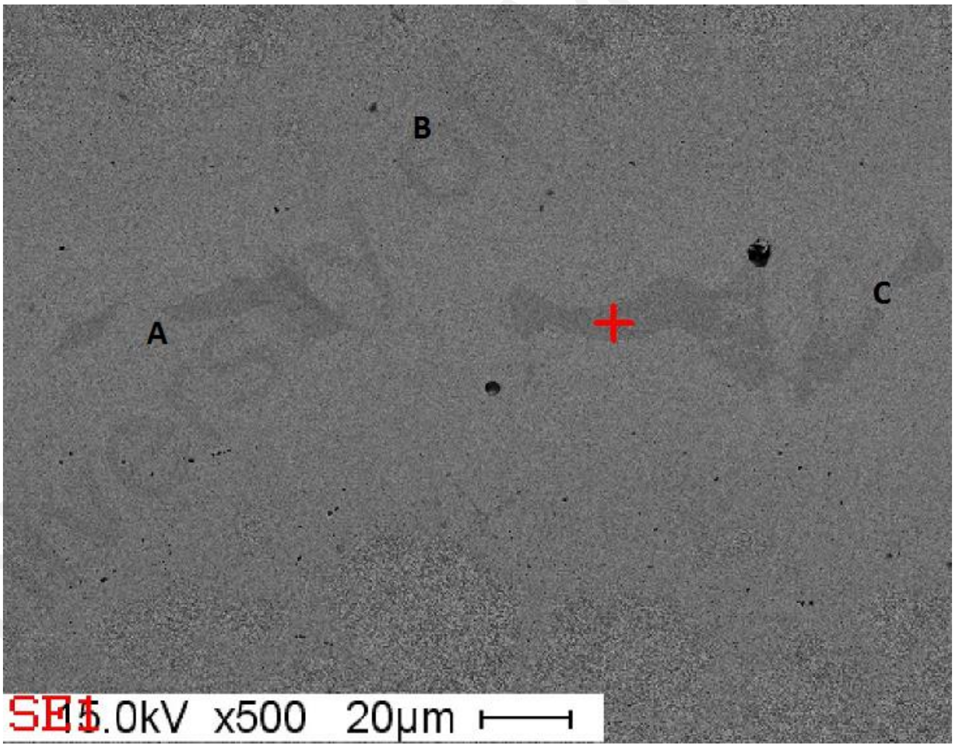


Figure 5.9: EDS image for optimum strength joints of brazed copper using filler metal of VZ2250

Element composition of braze layer show in Table 5.1 show that point A have highest composition of phosphorus (14.65 % wt) with less nickel (6.61 %wt). Tin element only detected at B point 2.47 % and C 6.63 %. Darker region as see from Figure 5.9 present phosphorus and tin element for brazed copper using Cu–7.0Ni-9.3Sn–6.5P filler metal. While at grey area from the image near copper substrate show present of most phosphorus element.

Table 5.1: Element composition brazed copper layer using of VZ 2250 filler metal

Point	Element composition (% wt)			
	P	**Cu**	**Ni**	**Sn**
A	14.65	78.71	6.61	-
B	1.10	84.62	11.81	2.47
C	0.92	88.4	4.46	6.63

Brazing copper using of Cu–5.7Ni–9.7Sn–7.0P (MBF2005) filler metal show the result as in Figure 5.10. The points for EDS analysis were marked as A, B and C. The sample for this inspection brazed at brazing temperature 680 °C held for 10 minutes. For this parameter the darker region A and C show the highest present of phosphorus and nickel. At point B near to copper substrate surface show less present of phosphorus and nickel.

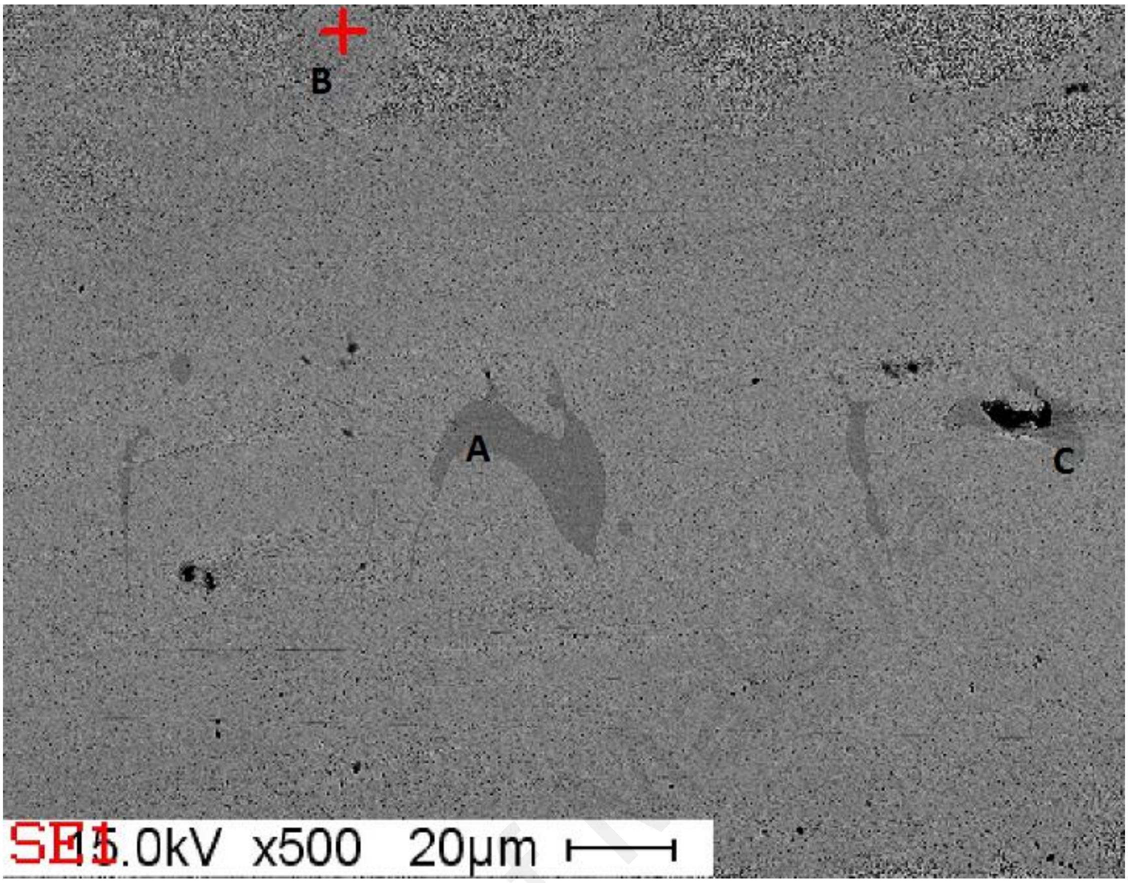


Figure 5.10: EDS result for highest strength brazed copper using filler metal of MBF 2005

From the optimum sample observed under FESEM and EDS analysis it show that P-rich phase appear for both brazed copper using of VZ2250 and MBF2005 filler metal. Also can see the P-rich phase concentrated in middle of joining with appear darker region. The present of tin detected just the end of outer darker region. This holding time 15 minutes for VZ filler and 10 minutes for MBF filler tin is fully diffused and solidified in copper, and achieving the eutectic phase.

Table 5.2: Element composition for brazed copper (optimum strength sample) using filler metal of MBF 2005

Point	Element composition (%wt)			
	P	**Cu**	**Ni**	**Sn**
A	15.34	80.76	3.90	-
B	1.23	97.1	1.66	-
C	1.38	93.57	1.64	3.41

CHAPTER 6: **CONCLUSION AND RECOMMENDATIONS**

6.1 Conclusion

Brazing pure copper (99.9 %) where joint copper substrate and copper upper part was set at certain parameters i.e temperature and holding time using alloy filler Cu–7.0Ni-9.3Sn–6.5P (VZ2250) and Cu–5.7Ni–9.7Sn–7.0P (MBF2005) show a significant effects to it morphology and joint strength of the joint. Some of it effect list as follow:

- Copper braze interface, joining of brazing is occurred by formation of diffusion layer at the area of joints.

- The brazing layer's microstructure show the changes by increment of brazing holding time

- The microstructure of copper braze join are varied with the brazing temperature. Increasing of temperature give nearly full solidification of filler.

- The joints strength effect by it brazing temperature, at higher temperature better joint strength when held at intermediate holding time.

The brazed copper filler metal of VZ2250 obtained exhibited maximum shear strength 148 MPa when brazed at 680 °C with holding time 15 minutes.

While brazed pure copper using filler metal of MBF 2005 show its maximum shear strength 168 MPa when braze at 680 °C with 10 minutes holding time.

From the research found that by prolonging the holding time, more copper substrate was dissolve into the filler alloy with higher brazing temperature.

6.2 Recommendations

Some recommendation may consider for future improvement of this research area. The suggestion to get better outcome includes:

- Materials selection – pure copper without any oxidation and surface layer preparation without any void. Cleans and good sample of material is one of requirement to get better quality of joints.

- Sample cutting – using linear cutting machine to make sure sample cutting symmetrical for best observation under OM and other microscopy apparatus. The straight cutting easy for grinding and polishing process. Copper is soft metal so the right cutting speed of cutter to be consider in order to avoid the hard scratch of the cutting surface. Cutter with sharp blade and constants speed also need to be care during cutting process.

- Surface clearing and clearness – for better observation of microstructure cutting surface of joints must be free from any scratch and dirt or avoids. Grinding process start with low speed for long time. The quality of high grade sand paper to be use during the process by use of 1500, 2000 and 4000 grade. Prolong grinding is better for surface clearness.

- Parameter of brazing – Need to be consider brazing temperature for further investigation by increasing the temperature range 700 °C- 800 °C. For holding time may consider for 20 - 30 minutes. By increasing these brazing parameter any major different effect to the joint strength may be seen.

- Tensile strength and hardness test – Mechanical properties of brazed copper need to get further strength test to see the effect of brazing parameter to the joint performances.

These brazing testing sample prepared by using vacuum electric furnace, meanwhile to have more strength performance microwave heating furnace can be consider for future investigation. The comparison within braze using two brazing method electric furnace and microwave furnace could be another gap to fill in this area.

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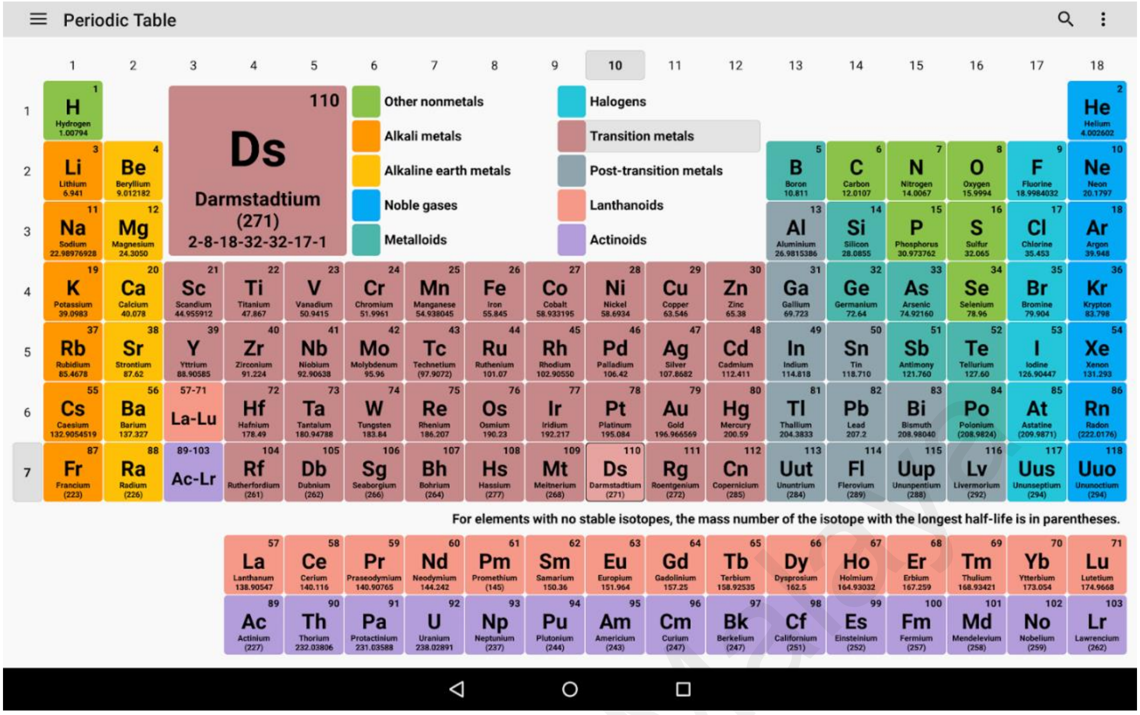
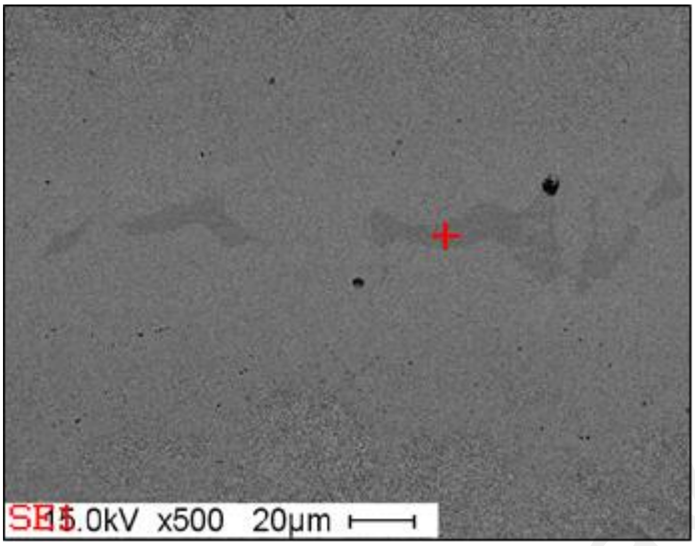
APPENDIX A – PROJECT GANTT CHART

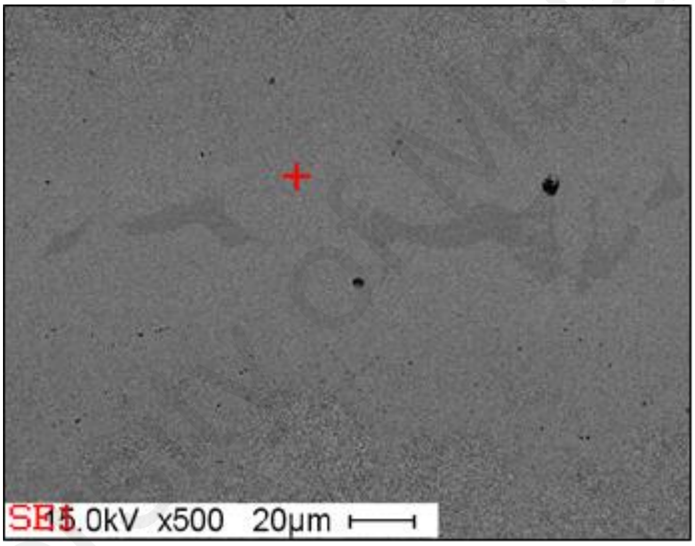
Item	Month	Aug-17				Sep-17				Oct-17				Nov-17				Dec-17			
	Week/Proc	1	es2	descrip3	tion 4	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
1	Approval for Project Extention																				
2	Preparing brazing material																				
3	Perform brazing sample																				
4	Perform grinding and polishing sample																				
6	Perform microstructure testing																				
7	Report writing																				
8	Submission research report																				
	Research Presentation																				

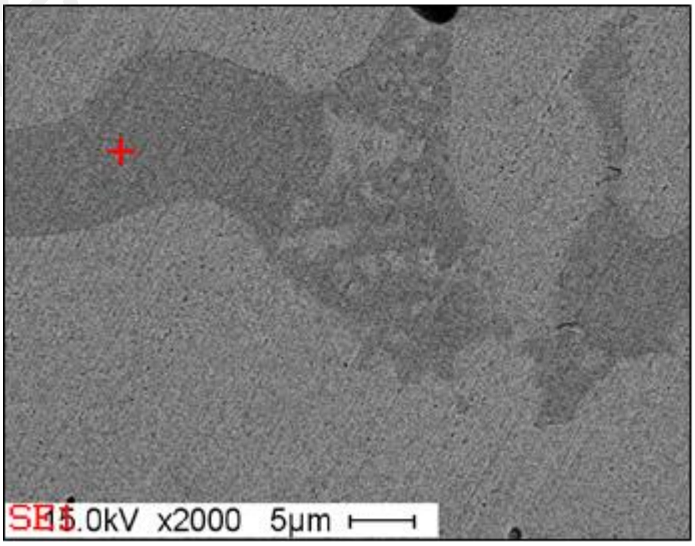
APPENDIX B – PERIODIC TABLE

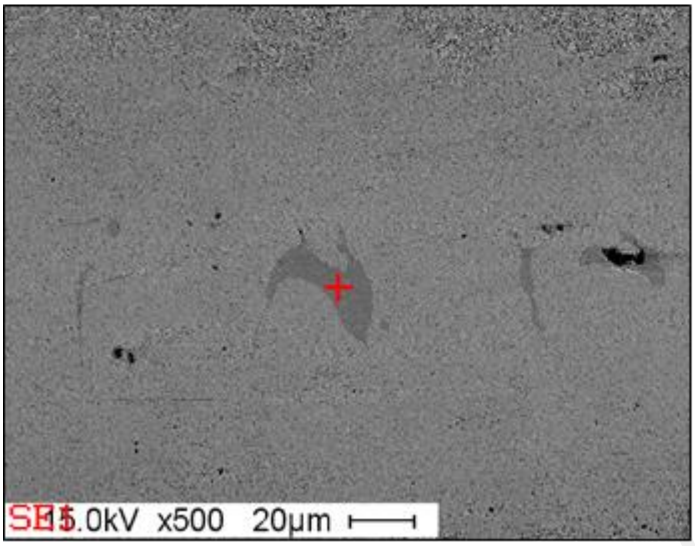


APPENDIX C SCANNING IMAGES





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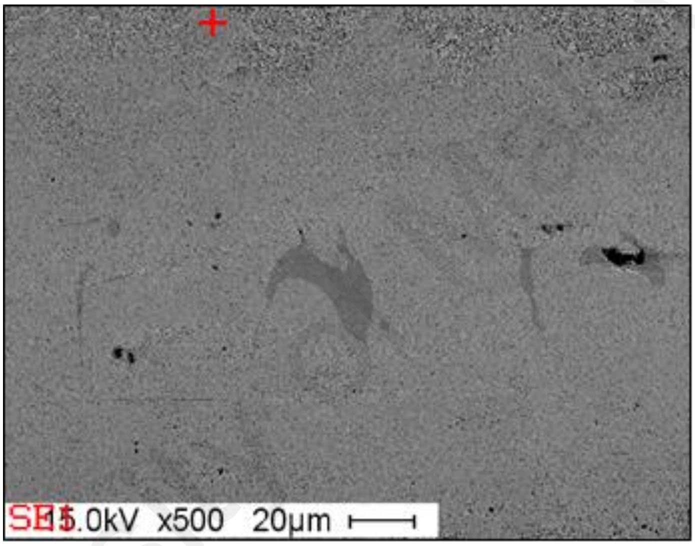
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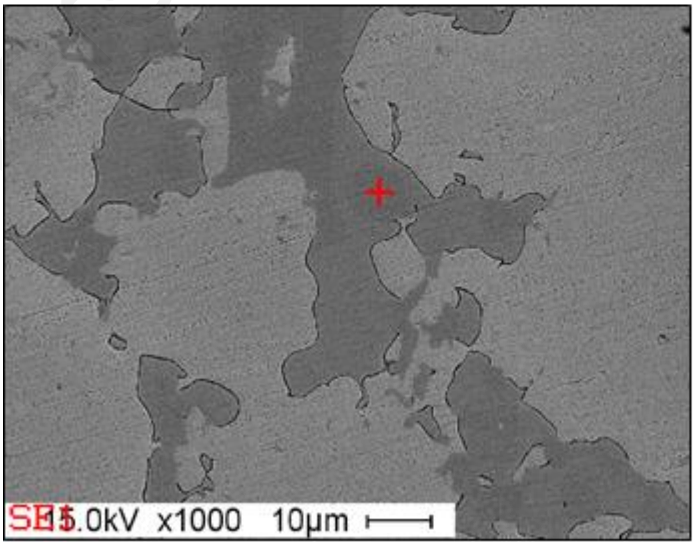
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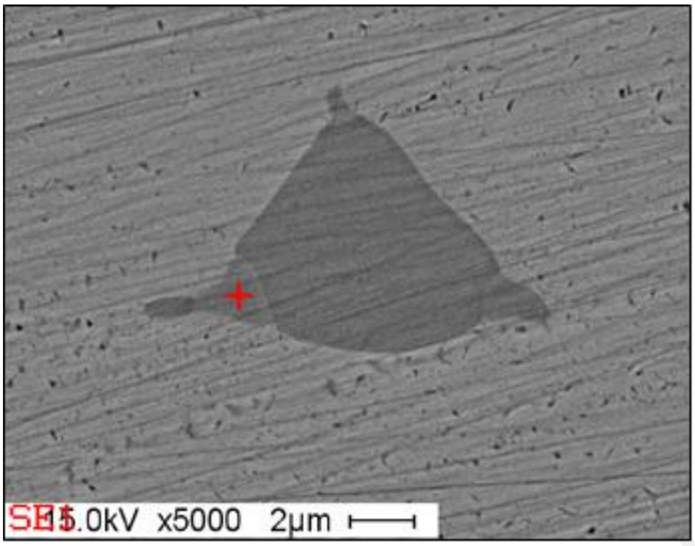
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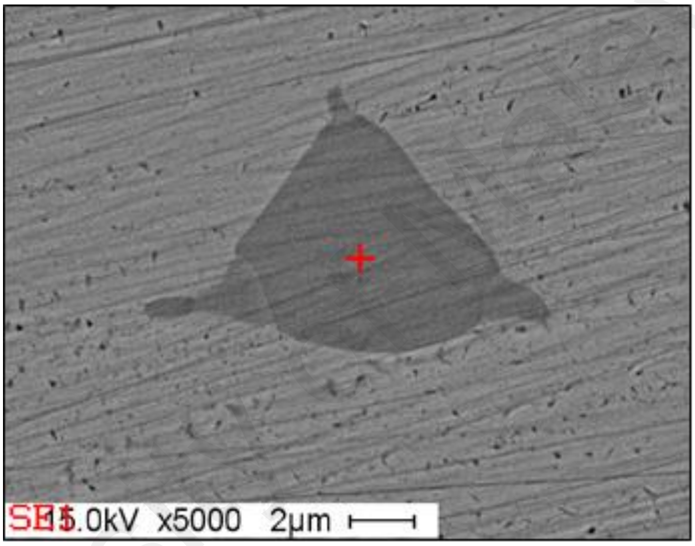
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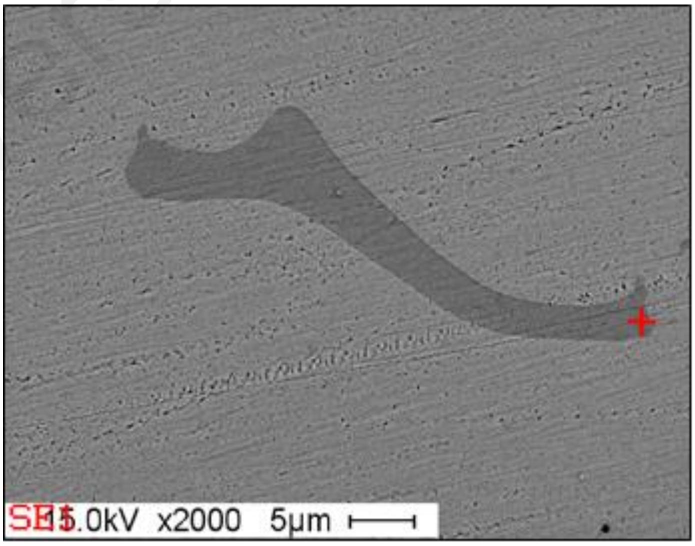
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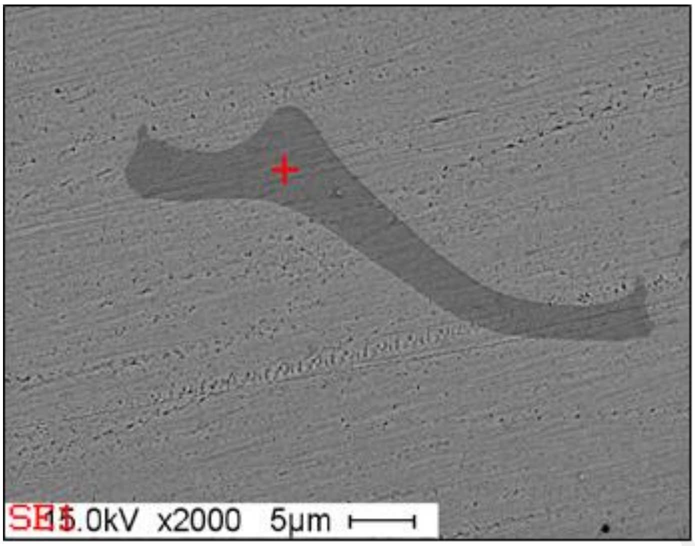
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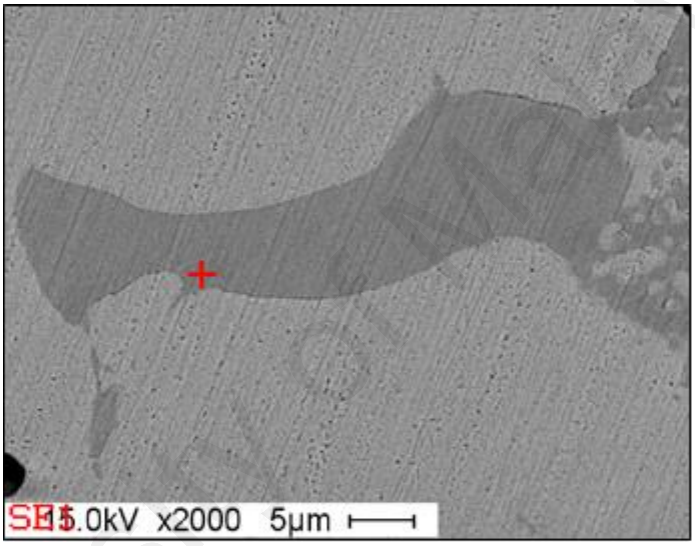
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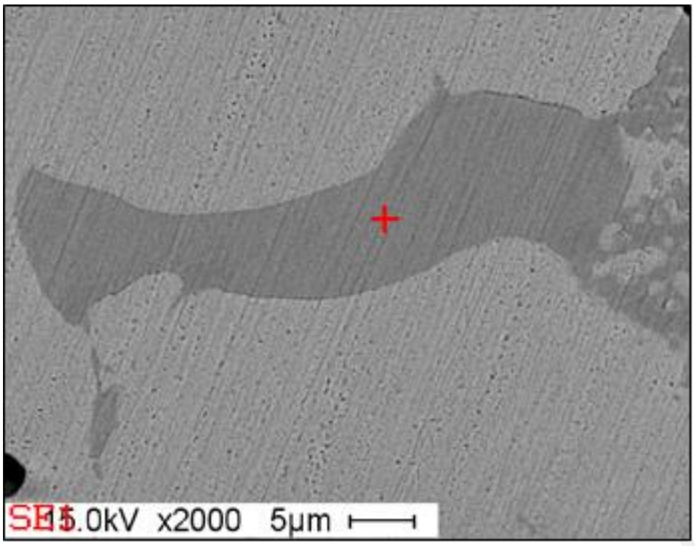
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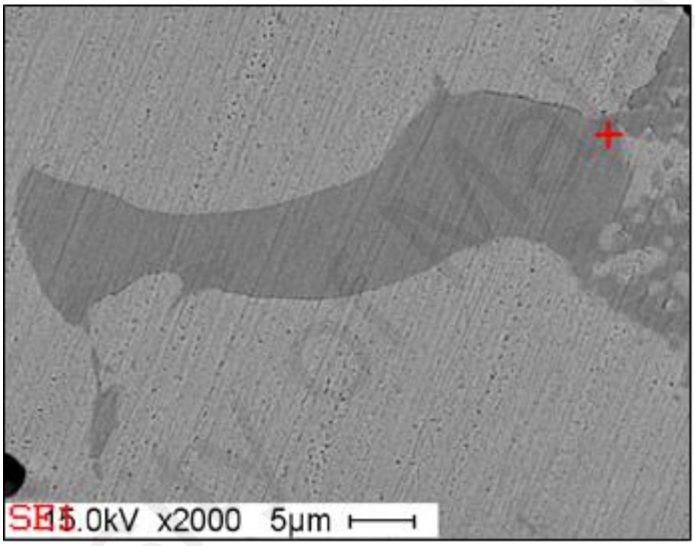
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