

**CHARACTERIZATION OF NANOCOOLANT FOR  
IMPROVING COOLING CHANNEL DESIGN IN  
HOT PRESS FORMING**

**(PENGAJIAN NANOCOOLANT UNTUK MEREKA  
BENTUK SALURAN ACUAN DALAM PROSES HOT  
PRESS FORMING)**

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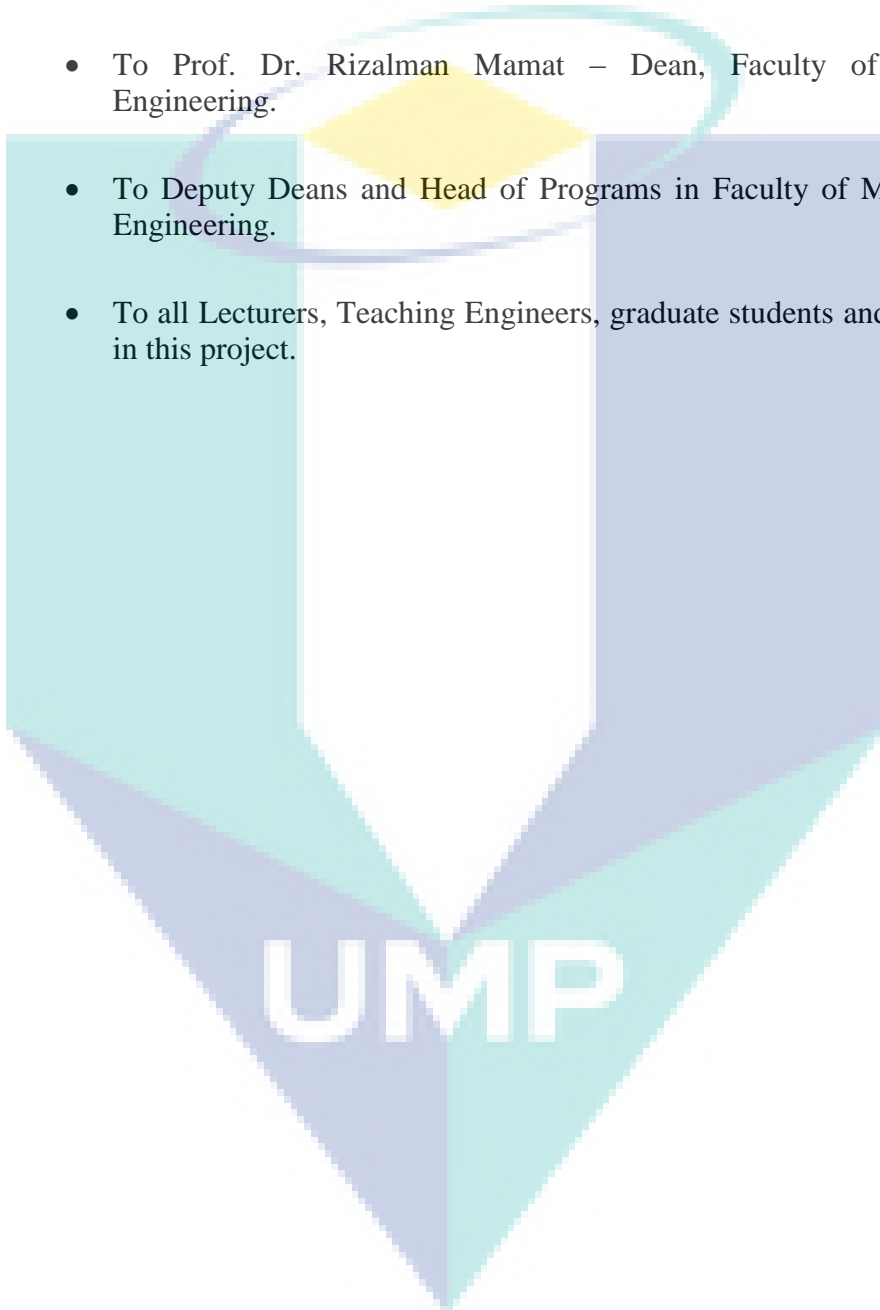
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## ABSTRACT

Hot press forming (HPF) to develop ultra-high strength steel (UHSS) of boron sheet metals for vehicle inner body panels offers efficient fuel consumption in order to reduce carbon dioxide gas emissions by weight reduction and improves passenger safety because of its high mechanical properties. To strengthen a UHSS material such as boron steel, it is required to manufacture from HPF process. The sheet metal is heated up to austenitic temperature and then rapidly quenched in an enclosure dies at a certain holding time to exhibit martensitic transformation phase. In the HPF process, a similar die is used as in the cold stamping operation, but with the additional cooling channel. Currently, water is used as coolant in the HPF process to quench boron steels in a closed die with a cooling channel. However, to enhance the performance of HPF dies and increase the mechanical properties of hot pressed boron steel, the fluid with better thermal properties will be used instead of normal water. During the quenching operation, an optimum cooling rate and homogeneous temperature distribution on hot blanks towards the achievement of the martensitic microstructure transformation as well as high mechanical properties. This study dispersed  $\text{Al}_2\text{O}_3$  nanoparticles with an average diameter of 13 nm in three volume percentage base ratios of water (W) to ethylene glycol (EG) such as 60%:40%, 50%:50%, and 40%:60% by using the two-step preparation method. The two main parameters in cooling rate performance are thermal conductivity and dynamic viscosity. The nanocoolant of  $\text{Al}_2\text{O}_3$ /water–ethylene glycol mixture is prepared for the volume concentration range of 0.2 to 1.0%. The thermal conductivity and dynamic viscosity are then measured at a temperature range of 15 to 55 °C. The heat transfer distribution of the heated blanks with nanocoolant and chilled water are determined by experimental analysis of HPF process. It was found that the highest enhancement of thermal conductivity was observed to be 10% higher than base fluid for 1.0% volume concentration at 55 °C in 60%:40% (W/EG). However, the highest enhancement of dynamic viscosity was measured to be 39% for 1.0% volume concentration in 40%:60% (W/EG) at 25 °C. The convective heat transfer coefficient of 1.0% concentration in 60%:40% (W/EG) at 25 °C is enhanced by 25.4% better than that of 50%:50% and 40%:60% (W/EG) base fluid. Therefore, this study recommends the use of  $\text{Al}_2\text{O}_3$  in 60%:40% (W/EG) mixture with volume concentration of less than 1.0% for application in cooling channel of HPF dies. It was also evident that the pattern of temperature distribution of the experimental results was in agreement with some researchers. The tensile strength and Vickers hardness values of the hot pressed parts were evaluated to be approximately 1550 MPa and 588 HV, respectively. In conclusion, nanocoolant as cooling fluid with higher convection heat transfer coefficient compared to the chilled water can reduce the cycle time and increase the productivity of HPF process.

(Keywords: Hot Press Forming, Thermal Conductivity, Dynamic Viscosity and Tensile Strength)

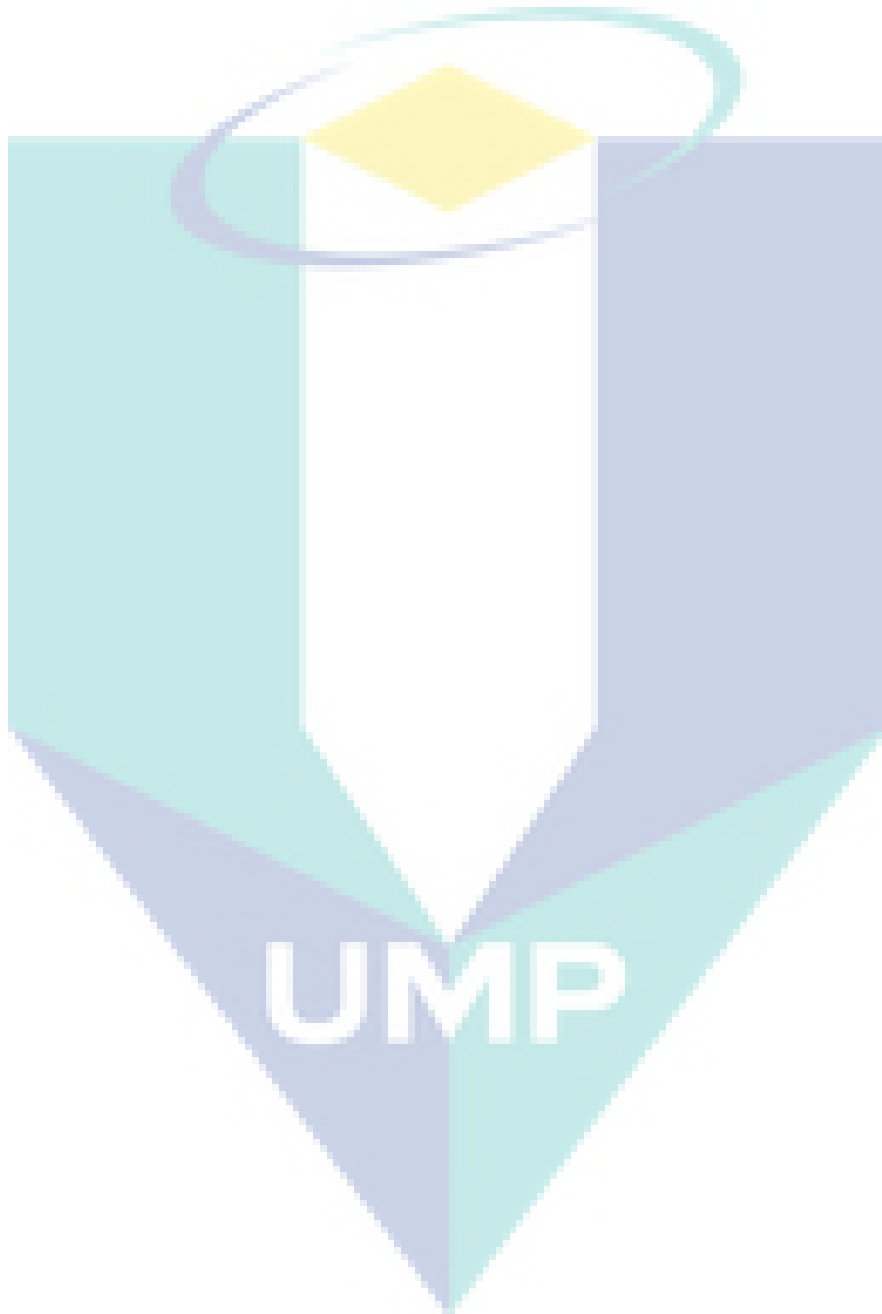
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## ABSTRAK

Hot press forming proses (HPT) untuk membangunkan UHSS boron keluli untuk panel badan dalaman kenderaan menawarkan penggunaan bahan api yang cekap untuk mengurangkan pelepasan gas karbon dioksida disebabkan oleh pengurangan berat badan dan meningkatkan keselamatan penumpang kerana sifat mekanikal yang tinggi. Boron keluli dipanaskan sehingga suhu austenitik dan kemudian disejutkan dengan cepat dalam sebuah acuan pada waktu pembajaan tertentu untuk mempamerkan fasa transformasi martensit. Dalam proses HPF, acuan yang sama digunakan seperti dalam operasi stamping sejuk, tetapi dengan saluran penyejukan tambahan. Pada masa ini, air digunakan sebagai penyejuk dalam proses HPF untuk menghilangkan keluli boron dalam acuan dengan saluran penyejukan. Walau bagaimanapun, untuk meningkatkan prestasi acuan HPF dan meningkatkan sifat mekanik keluli boron ditekan panas, bendalir dengan sifat haba yang lebih baik akan digunakan dan bukannya air biasa. Semasa operasi pelindapkejutan, kadar penyejukan optimum dan pengagihan suhu homogen pada kekosongan panas ke arah pencapaian transformasi mikrostruktur martensitik serta sifat mekanikal yang tinggi. Kajian ini menyebarkan nanopartikel  $Al_2O_3$  dengan diameter purata 13 nm dalam tiga nisbah peratusan air kepada etilena glikol (EG) seperti 60%:40%, 50%:50%, dan 40%:60% oleh menggunakan kaedah penyediaan dua langkah. Kedua-dua parameter utama dalam prestasi kadar penyejukan adalah kekonduksian haba dan kelikatan dinamik. Nanocoolant campuran  $Al_2O_3$ /air-etilena glikol disediakan untuk kepekatan isipadu 0.2 hingga 1.0%. Kekonduksian haba dan kelikatan dinamik kemudian diukur pada julat suhu 15 hingga 55 °C. Analisis haba dilakukan untuk memastikan pengedaran suhu homogen yang dipanaskan. Data simulasi kemudian dibandingkan dengan penemuan eksperimen untuk tujuan pengesahan. Telah didapati bahawa peningkatan kekonduksian haba tertinggi adalah 10% lebih tinggi daripada bendalir asas untuk kepekatan isipadu 1.0% pada 55 °C dalam 60%:40% (W/EG). Walau bagaimanapun, peningkatan kelikatan dinamik yang tertinggi diukur sebanyak 39% untuk kepekatan isipadu 1.0% dalam 40%:60% (W/EG) pada 25 °C. Koefisien pemindahan haba konveksi kepekatan 1.0% dalam 60%:40% (W/EG) pada 25 °C ditingkatkan dengan 25.4% lebih baik daripada 50%:50% dan 40%:60% (W/EG) cecair. Oleh itu, kajian ini mengesyorkan penggunaan  $Al_2O_3$  dalam campuran 60%:40% (W/EG) dengan kepekatan isipadu kurang daripada 1.0% untuk aplikasi dalam saluran penyejukan pembentukan tekanan panas. Ia juga terbukti bahawa corak pengedaran suhu model analisis unsur terhingga bersesuaian dengan hasil eksperimen. Kekuatan tegangan dan nilai kekerasan Vickers bahagian yang ditekan panas dinilai masing-masing kira-kira 1550 MPa dan 588 HV. Sebagai kesimpulan, nanocoolant sebagai cecair penyejuk dengan pekali pemindahan haba konveksi yang lebih tinggi berbanding dengan air sejuk boleh mengurangkan masa kitaran dan meningkatkan produktiviti proses pembentukan panas

## TABLE OF CONTENT

<b>DECLARATION</b>	
<b>TITLE PAGE</b>	
<b>ACKNOWLEDGEMENTS</b>	<b>ii</b>
<b>ABSTRACT</b>	<b>iii</b>
<b>ABSTRAK</b>	<b>v</b>
<b>TABLE OF CONTENT</b>	<b>vi</b>
<b>LIST OF TABLES</b>	<b>x</b>
<b>LIST OF FIGURES</b>	<b>xi</b>
<b>LIST OF SYMBOLS</b>	<b>xiv</b>
<b>LIST OF ABBREVIATIONS</b>	<b>xv</b>
<b>CHAPTER 1 INTRODUCTION</b>	<b>1</b>
1.1 Background of Study	1
1.2 Problem Statement	4
1.3 Objectives	6
1.4 Scope of Study	6
1.5 Hypothesis	8
1.6 Thesis Organisation	8
<b>CHAPTER 2 LITERATURE REVIEW</b>	<b>11</b>
2.1 Introduction	11
2.2 Background of Nanofluids as Coolant	11
2.3 Properties of Water-Ethylene Glycol Based Nanocoolants	13

2.4	Nanoparticles as Suspended Material	15
2.4.1	Nanoparticle materials	18
2.4.2	Types of synthesized nanofluids	19
2.5	Nanofluids Preparation	20
2.6	Thermal Properties of Nanofluids	22
2.6.1	Dynamic viscosity	23
2.6.2	Thermal conductivity	25
2.7	Sheet Metal of Boron Steel	27
2.8	Hot Press Forming Process	29
2.8.1	Heating operation	32
2.8.2	Forming operation	36
2.8.3	Punching and loading force	39
2.9	Heat Transfer Mechanism	41
2.9.1	Surface condition	41
2.9.2	Fluid flow types	42
2.9.3	Tools temperature and contact pressure	43
2.10	Cooling Channel System of Die Design	45
2.11	Summary	47
<b>CHAPTER 3 NANOCOOLANT PREPARATION, CHARACTERISATION AND AUGMENTATION</b>		<b>49</b>
3.1	Introduction	49
3.2	Materials and Preparation of Nanocoolants	51
3.3	Thermal Conductivity Measurement	54
3.4	Dynamic Viscosity Measurement	57
3.5	Results and Discussion	59

3.5.1	Thermal conductivity of nanocoolant	59
3.5.2	Dynamic viscosity of nanocoolant	63
3.5.3	Heat transfer coefficient of nanocoolant	68
3.6	Summary	71
<b>CHAPTER 4 EXPERIMENTAL INVESTIGATIONS OF NANOCOOLANT APPLICATION IN HOT PRESS FORMING PROCESS</b>		<b>73</b>
4.1	Introduction	73
4.2	Sample Preparation	75
4.2.1	Nanocoolants as cooling fluid preparation	76
4.3	Thermocouples Setup	78
4.4	Hot Press Forming of Boron Steel Sheet Metal	79
4.4.1	Experimental study of temperature distribution	82
4.4.2	Tensile test measurements	84
4.4.3	Micro-hardness measurements	86
4.4.4	Metallographic observation study	87
4.5	Thermal Analysis of Nanocoolant and Chilled Water in Hot Press Forming	89
4.6	Microstructural Transformation Analysis of Hot Pressed Boron Steel	91
4.7	Tensile Strength Analysis of Hot Pressed Boron Steel	95
4.8	Hardness Analysis of Hot Pressed Boron Steel	97
4.9	Summary	100
<b>CHAPTER 5 CONCLUSION</b>		<b>101</b>
5.1	Conclusion	101
5.2	Contributions to Knowledge	103
5.3	Future Works	104
<b>REFERENCES</b>		<b>106</b>



<b>APPENDIX A G-code for cutting tensile test specimen from hat-shaped part</b>	<b>123</b>
<b>APPENDIX B Heat transfer coefficient values of nanocoolant</b>	<b>124</b>
<b>APPENDIX B1 Sample of cooling rate for sheet metal blank</b>	<b>125</b>
<b>APPENDIX C Sedimentation observation of aluminium oxide/water-eg mixture after a month of preparation</b>	<b>126</b>
<b>APPENDIX D Sample of tensile strength and hardness measurement</b>	<b>128</b>
<b>APPENDIX E International Communications in Heat and Mass Transfer</b>	<b>129</b>
<b>APPENDIX F Joint Conference IMEC &amp; APCOMS 2015</b>	<b>130</b>
<b>APPENDIX G International Conference on Nanoscience and Nanotechnology 2018</b>	<b>131</b>
<b>List of publication</b>	<b>132</b>

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## LIST OF TABLES

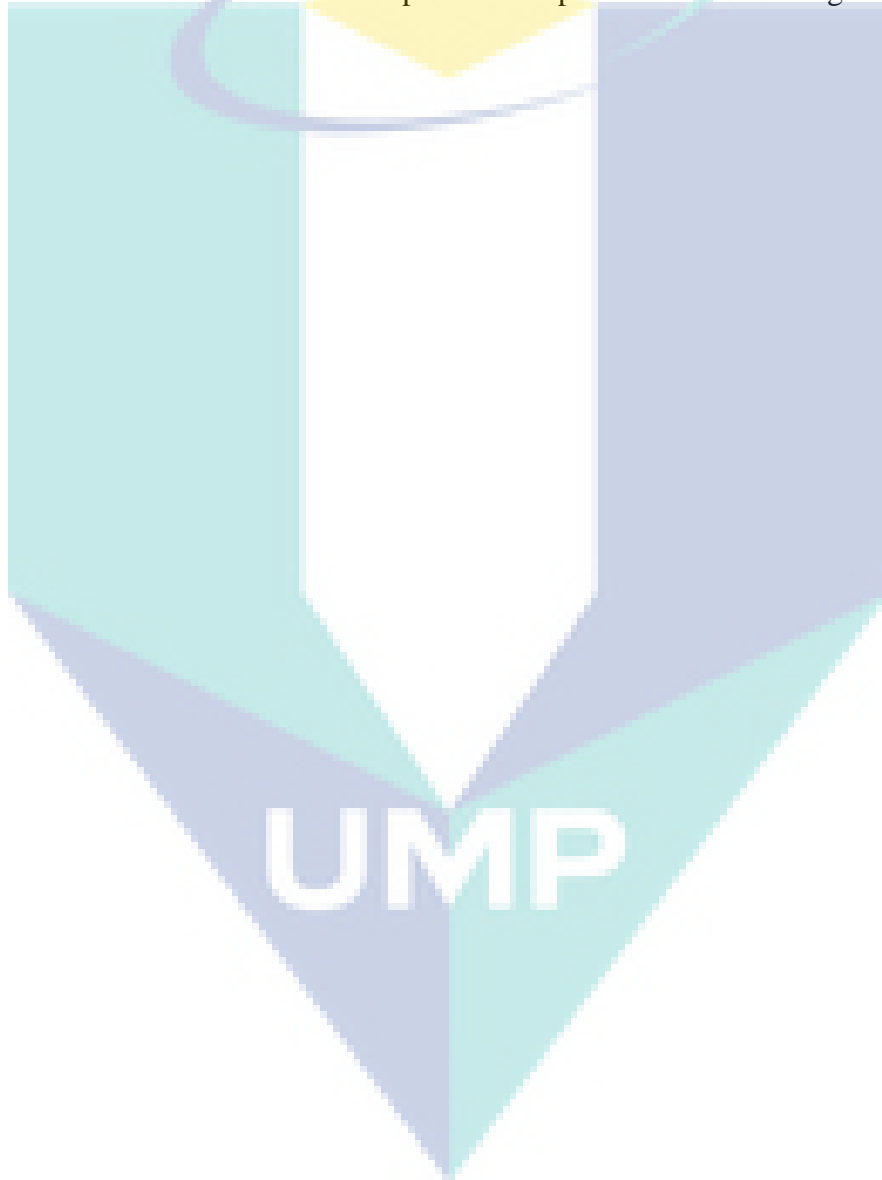
Table 2.1	Thermo-physical property of different types of metal and liquid	13
Table 2.2	Various types of nanoparticles and micrograph images	17
Table 2.3	Types of nanoparticles dispersed in nanofluids	20
Table 2.4	Dynamic viscosity investigations for different types of nanocoolants	24
Table 2.5	Thermal conductivity study for diverse types of nanofluids	26
Table 2.6	Chemical compositions of boron steel weight percentage and mechanical properties before and after quenching operation	28
Table 2.7	Thermal-physical properties of boron steel	29
Table 2.8	Stamping process in hot temperature conditions	40
Table 2.9	Results of contact pressure and standard deviation of HTC	44
Table 3.1	Properties of nanoparticles used in experiment	52
Table 3.2	Properties of Ethylene Glycol solution	53
Table 3.3	Thermal conductivity models for nanocoolant	56
Table 3.4	Dynamic viscosity models for nanocoolant	58
Table 4.4	The features of mechanical press machine model OCP 80	80
Table 4.5	The features of hydraulic press machine	81
Table 4.6	Specifications of Universal Tensile Machine	85
Table 4.7	Specifications of Wilson Vickers 402 MVD machine	87
Table 4.8	Specification of LOM, Olympus BX51M machine	88
Table 4.9	Temperature distribution for HPF tools and heated blank	89
Table 4.10	Micrographs of boron steel blank with several quenching time periods	92
Table 4.10	Micrographs of boron steel blank with several quenching time periods (Continued)	93
Table 4.11	Tensile strength value for several specimens of hot pressed boron steel	96
Table 4.12	Hardness value for several specimens of hat-shaped boron steel	98

## LIST OF FIGURES

Figure 1.1	Tensile strength between UHSS and typical sheet metal	2
Figure 1.2	Mechanical properties of boron steel before and after hot forming process	3
Figure 1.3	Thesis organisation	10
Figure 2.1	Freezing point and boiling point of water-ethylene glycol mixture	14
Figure 2.2	One-step method technique	21
Figure 2.3	Two-step method technique	22
Figure 2.4	Graph of velocity pattern versus velocity	23
Figure 2.5	Temperature, time and transformation diagram of boron steel at various cooling rates	28
Figure 2.6	Direct hot forming process	30
Figure 2.7	Indirect hot press forming	31
Figure 2.8	Hat-shape profile bending operation	32
Figure 2.9	Various types of heating system (a) Roller hearth furnace, (b) Induction heating and (c) Electrical resistance heating	33
Figure 2.10	Induction heating operation (a) Schematics diagram of the customized induction furnace and (b) Variation of temperature curve as function of time for several feeding speed	35
Figure 2.11	Localize heating (a) Positioning two rectangular electrodes on the blank and the temperature distribution diagram; and (b) Result of punching load for different heating temperatures in shearing region	36
Figure 2.12	Forming Limit Diagram of B-pillar	37
Figure 2.13	Simulation of B-pillar in hot press forming process	37
Figure 2.14	Geometries of tools and steel sheet for hat-shape and V-shape	38
Figure 2.15	Degree of springback after V-shape bending with the relationship of temperature in (a) Bending region; and (b) Flange region	39
Figure 2.16	Hot forming process of UHSS sheet metal by using resistance heating	40
Figure 2.17	Relationship between the maximum stamping load and heating temperature of UHSS sheet metal	40
Figure 2.18	Thermal contact resistance (a) Ideal thermal contact; (b) Actual thermal contact	42
Figure 2.19	Experimental setup and instrument for heat transfer coefficient testing	44
Figure 2.20	Relation between HTC as function of contact pressure for different tool temperatures	45
Figure 2.21	Schematic diagram of hot press forming tool design	46

Figure 2.22	Schematic diagram of the quenching tool integrated with hot forming operation	47
Figure 3.1	Flow chart of nanocoolant characterisation and experimental convection heat transfer	51
Figure 3.2	The Al <sub>2</sub> O <sub>3</sub> nanoparticles with size of 13 nm	52
Figure 3.3	FESEM result of dry Al <sub>2</sub> O <sub>3</sub> nanoparticles at X300,000 magnification	52
Figure 3.4	Al <sub>2</sub> O <sub>3</sub> nanocoolant immersed in ultrasonic bath heater	54
Figure 3.5	Nanocoolant samples of Al <sub>2</sub> O <sub>3</sub> /ethylene glycol-water mixtures after a month of preparation	54
Figure 3.6	(a) Schematic diagram of thermal conductivity measurement; (b) Experiment setup for thermal conductivity measurement.	56
Figure 3.7	(a) Schematic diagram of dynamic viscosity measurement; (b) Experiment setup for dynamic viscosity measurement	58
Figure 3.8	Variation of thermal conductivity ratio as function of nanoparticles volume concentrations in W/EG mixture at 25 °C	60
Figure 3.9	Thermal conductivity of different nanoparticle concentrations for three different weight of W/EG mixture base fluids	63
Figure 3.10	Variation of viscosity ratio as fraction of nanoparticle volume concentrations in W/EG mixture at 25 °C	64
Figure 3.11	Viscosity of different nanoparticle concentrations for three different weight of water-ethylene glycol mixture base fluids	68
Figure 3.12	Relationship between thermal conductivity as function of nanoparticles volume concentration for three different base fluids	69
Figure 3.13	Distribution of heat transfer coefficient of 60%:40% water-ethylene glycol mixture based Al <sub>2</sub> O <sub>3</sub> nanocoolant	71
Figure 4.1	Flow chat of experimental thermal analysis and application in HPF	74
Figure 4.2	Pre-forming process of hat-shaped samples	75
Figure 4.3	The preparation of Al <sub>2</sub> O <sub>3</sub> nanocoolant	77
Figure 4.4	Thermocouples location in HPF tool a) Schematic diagram b) Three thermocouples in upper tool	79
Figure 4.5	Fabrication of hat-shape sample by using mechanical press machine, OCP 80	81
Figure 4.6	Hydraulic press machine used in hot forming operation	81
Figure 4.7	The experimental equipment setup for hot press forming tool	82
Figure 4.8	Hot press forming process sequence of hat-shaped blank	83
Figure 4.9	Location of hot formed samples for tensile specimen, hardness test and microstructure analysis	85
Figure 4.10	Specimen for tensile strength test	85
Figure 4.11	Tensile strength measurement with Universal Tensile Machine	86

Figure 4.12	Hardness measurement with Vickers Micro-hardness Machine, Wilson Vickers 402 MVD	87
Figure 4.13	Microstructural analysis by using light optical microscopy machine	88
Figure 4.14	Heat transfer distribution between nanocoolant and chilled water for hat-shaped tool	90
Figure 4.15	Ultimate tensile strength of blank samples at several cooling conditions	96
Figure 4.16	Vickers hardness reading HV20 with diamond shaped indent 50 $\mu\text{m}$	98
Figure 4.17	Hardness value of hot pressed samples at several cooling conditions	99



## LIST OF SYMBOLS

MPa	Mega Pascal
min	Minutes
s	Second
$^{\circ}\text{C}\cdot\text{s}^{-1}$	Degree Celsius per Second
mm	Millimeter
$\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$	Watt per Meter Kelvin
$E$	Elastic of Modulus
$k$	Thermal Conductivity
$k_{bf}$	Thermal Conductivity of Base Fluid
$k_{nf}$	Thermal Conductivity of Nanocoolant
$\mu$	Dynamic Viscosity
$\mu_{nf}$	Dynamic Viscosity of Nanocoolant
$\mu_{bf}$	Dynamic Viscosity of Base Fluid
$C_p$	Specific Heat of Particle
$C_{bf}$	Specific Heat of Base Fluid
$C_{nf}$	Specific Heat of Nanocoolant
$\varphi$	Volume Fraction
$\phi$	Volume Concentration
$\phi_1$	Initial Volume Concentration
$\phi_2$	Final Volume Concentration
$\rho$	Density
$\rho_{bf}$	Density of Base Fluid
$\rho_{nf}$	Density of Nanocoolant
$\rho_p$	Density of Particle
$\omega$	Weight Concentration
$Re$	Reynolds Number
$Pr$	Prandtl Number
$Pr$	Prandtl Number of Nanocoolant
$Nu$	Nusselt Number
$h$	Heat Transfer Coefficient
$q$	Heat Transfer
$A$	Heat Transfer Area
$T_f$	Surrounding Fluid Temperature
$T_s$	Surface Temperature
$T_b$	Bulk Temperature
$t$	Thickness
$B$	Bending Length
$P$	Loading Force
$L$	Length
$V$	Velocity
$D$	Distance
BR	Base Ratio

## LIST OF ABBREVIATIONS



AHSS	Advanced High Strength Steel
Al <sub>2</sub> O <sub>3</sub>	Aluminium Oxide
CuO	Copper Oxide
FCNT	Functionalised Carbon Nanotube
FEA	Finite Element Analysis
FESEM	Field Emission Scanning Electron Microscopy
FLD	Forming Limit Diagram
HPF	Hot Press Forming
HSS	High Strength Steel
HTC	Heat Transfer Coefficient
HTCS	High Thermal Conductivity Tool Steel
IUPAC	International Union of Pure and Applied Chemistry
MWCNT	Multi-walled Carbon Nanotube
SEM	Scanning Electron Microscopy
SiC	Silicon Carbide
SiO <sub>2</sub>	Silicon Oxide
SKD	Hot Work Tool Steel
TEM	Transmission Electron Microscopy
UHSS	Ultra-High Strength Steel
EG	Ethylene Glycol
W	Water
ASHRAE	American Society of Heating, Refrigerating and Air Conditioning Engineers
BR	Base Ratio
ASTM	American Society for Testing and Materials International
LOM	Light Optical Microscopy
UTM	Universal Tensile Machine
DIN EN ISO	German Institute European Standard

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of Study

The increasing awareness of environmental pollution caused by vehicle emission has driven automotive manufacturers around the world to improve fuel efficiency by producing a lighter vehicle without compromising vehicle safety. Reducing the weight of the vehicle requires the component to have thinner material, but at the same time, it should not discredit the mechanical properties. This has resulted in the introduction of ultra-high strength steel (UHSS) materials. The development of UHSS material has paved the reduction in gas emission to the environment, energy saving and the production of safer vehicles. UHSS material has a higher tensile strength and lower weight ratio when compared to the mild steel (Karbasiyan et al., 2010). Most of the UHSS materials are having high mechanical properties as shown in Figure 1.1.

In conventional cold press forming process, the issues of poor formability, forming accuracy and greater springback have often occurred in high strength steel (HSS), advanced high strength steel (AHSS) and UHSS, respectively (Atlan, 2012). Hence, to overcome these problems of high strength steel forming, researchers and industries are actively developing hot press forming (HPF) technologies (Turetta, 2008). HPF process is a new forming method that can significantly enhance the formability of UHSS. In addition to high strength and dimensional accuracy of steel sheet formed, it can further avoid the springback of UHSS from cold forming process and achieve the purpose of weight reduction (Sever et al., 2012; Thanadgarn et al., 2013). Furthermore, UHSS material such as boron steel gains its final strength through the HPF process as heat treatment, which can increase the hardness and mechanical strength of boron steel up to 1400 MPa (Atlan, 2012). The obtained strength is two-fold



higher than the boron steel in annealed condition. As the capability of having an ultimate tensile strength of 1400 MPa and the possibility of weight reduction. Automotive industry implements the hot pressed parts as vehicle components such as the chassis, A-pillar, B-pillar, tunnel, bumper, roof rail and many others being formed by boron steel.

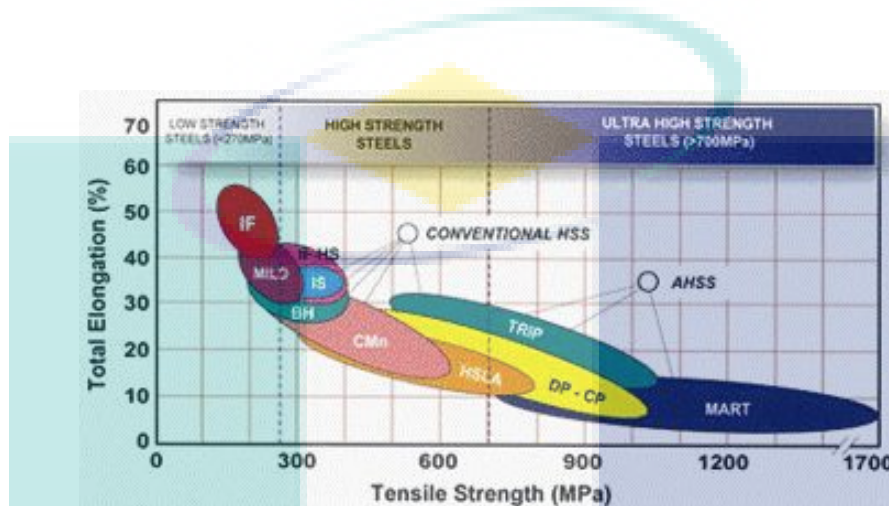


Figure 1.1 Tensile strength between UHSS and typical sheet metal  
Source: Atlan, 2012

Naderi, (2007) reported that boron steel, 22MnB5 produced a fully martensitic microstructure after using HPF process. Figure 1.2 shows the boron steel has a tensile strength of approximately 600 MPa at the initial state. Higher ultimate tensile strength can be attained by a rapid cooling of the hot forming tool at the cooling rate of at least  $27 \text{ K}\cdot\text{s}^{-1}$  (Merklein et al., 2008). Before HPF process, the boron steel consists of ferrite-pearlite microstructure must be austenitized in order to increase the elongation which is practical for the press forming operation. However, the yield strength of the boron steel is reduced during the austenite transformation phase. Finally, the martensitic transformation will transpire if the austenite cools immediate during the HPF process (Karbasiyan et al., 2010).

The HPF process also named as a press hardening process, which consists of three phases such as heating the sheet metal blank, press forming operation and part quenching (So et al., 2012). In the process, UHSS blank is cut into the rough dimension, and the blank is heated up to the required temperature of  $900 \text{ }^\circ\text{C}$  for 5 mins inside the furnace. Then, the heated blank is quickly transferred to the press to avoid the temperature of the blank cooled down in the atmosphere before forming operation.

Subsequently, the part is formed and cooled simultaneously by the water-cooled die for approximately 10 s. Due to the contact between the cool HPF dies and heated blank, the blank is cooled in the enclosure tools (Karbasiyan et al. 2010). Besides that, HPF process exists in two different methods that are the direct HPF and indirect HPF method. For direct HPF process, a blank is heated up in a furnace, transferred to the press and subsequently formed and quenched in the enclosure tool. While, for indirect HPF process, before the blank is austenitized inside the furnace, it has to perform the cold pre-forming operation. In this research project, the indirect HPF process was chosen due to the ability to install the heating element inside the hot forming tool.

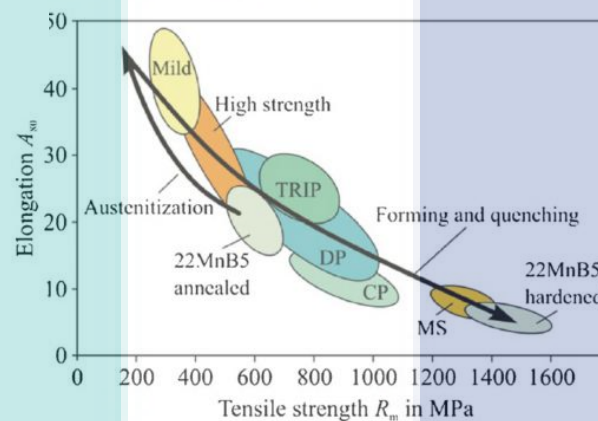


Figure 1.2 Mechanical properties of boron steel before and after hot forming process

Source: Karbasiyan et al. (2010)

The quenching operation during the HPF process does influence not only the cost effectiveness of the process, but also the final properties of the product. The purpose of the cooling channel system is to quench the hot specimen effectively and to achieve the cooling rate of at least  $27 \text{ K}\cdot\text{s}^{-1}$  during the martensitic transformation. The HPF dies cooling system provides the fluid coolant that flows through the cooling channel around the contours of the part. The heat flow in the hot pressed part depends on the heat transfer from the specimen to the HPF dies, the thermal conductivity within the dies, and the heat transfer from the dies to the coolant. The thermal conductivity and heat transfer within the tool and coolant can be considerably influenced by the types of cooling fluid and thermal cooling system.

Forced convection is the mechanism of heat transfer in most of the thermal cooling system or cooling system in engineering practice, and few such examples include automobile radiators and industrial power plant cooling system. Forced convection defines as to drive the fluid motion in the process of transferring the heat between mediums. The significant challenges involved in thermal engineering are to find approaches to reduce the heat transfer to a minimum or increase the heat to a maximum value. Pioneer researchers subjected to the nanocoolant field, such as Masuda et al. (1993) and Choi (1995) have successfully encountered new superior fluid called nanocoolants that has the ability to transfer heat as good as conventional heat transfer fluids or even better. Thus, the development of nanocoolant technology has expanded considerably over the past several years after the pioneer research made by Choi (1995) in the early 90's and is still growing until today.

The production of water base nanocoolants has been widely employed in many kinds of research over the past years. Several kinds of research have been reported on the experimental studies of nanocoolant in mixture (Kulkarni et al., 2008; Bayat and Nikseresht, 2012). Kulkarni et al. (2008) conducted an investigation on forced convection heat transfer for the base fluid mixture which composed of water and ethylene glycol in ratio of 40%:60%, where the enhancement was found to be 16%. Bayat and Nikseresht (2012) demonstrated that the enhancement of the average heat transfer coefficient was more significant in higher concentrations and higher Reynolds number by using 40%:60% water-ethylene glycol mixture. The nanocoolant as a heat transfer fluid is essentially determined through the heat transfer coefficient. While, the efficiency of the nanocoolant is evaluated from the heat transfer parameters, such as the Prandtl number, Nusselt number, and heat transfer coefficient (Bhanvase et al., 2014).

## **1.2 Problem Statement**

Hot press forming (HPF) process requires rapid cooling of the sheet metal blank inside the enclosure dies, and this includes the cooling system to be integrated with the hot forming tools to control the cooling rate. The cooling system must be designed to cool efficiently to achieve the maximum cooling rate and homogeneous temperature distribution over the hot pressed products. In cooling channel development, the effect of

heat transfer, hot forming tool materials and blank materials must be considered. At the same time, it must fulfil the required process system regarding to the heating temperature to press forming operation, transmitting time and heating furnace. Thus, one of the significant factors to be considered during the process is the design of the cooling channel, including the size, location and distribution temperature (Zhong et al., 2010; Liu et al., 2013).

Current practice in a local industry (Miyazu (M) Sdn. Bhd.), the mean for holding time during quenching is 12 s, and it takes about 25 to 30 s to completely produce one single part with a predicted  $27 \text{ K}\cdot\text{s}^{-1}$  of cooling rate. The cooling fluids that have been used in the HPF process are water or chilled water with the thermal conductivity of approximately  $0.5 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  (Lin et al., 2014). The side beam hot forming die is segmented into 12 blocks due to the cooling channel drilling process capability, with a limitation of 400 mm in length (Hafizuddin, 2014). The diameter of the cooling channel is 8 to 12 mm, the distance to loading surface is 10 to 12 mm, and the pitch is 10 mm. Therefore, dealing with ultra-high strength steel (UHSS) properties in HPF process, it requires the tool to cool down the blank immediately, for that a cooling system must be integrated into the hot forming tools.

Besides optimizing the cooling channel design and tool material, researchers are working to optimize the thermal conductivity with minimum cooling channel design by implementing nanocoolants to replace water and chilled water as cooling fluids in HPF process. This research project intends to increase the efficiency of thermal conductivity in order to enhance the convection heat transfer values by utilizing the nanocoolants, and to report a comparative study between conventional coolants and nanocoolants. There are several studies reported on the introduction of nanocoolants in the automotive field. Sharma et al. (2013) proposed a nanocoolant as engine based coolant and showed the improvement of thermal physical properties over the conventional fluids. The mixture of nanocoolant is based on the synthesizing of  $\text{Al}_2\text{O}_3$  with base fluids. The results indicated that the thermal conductivity and dynamic viscosity increases with the increase of volume concentration. Therefore, the fluids with better thermal properties will be used instead of normal water to enhance the cooling performance of the HPF dies, thereby providing the optimum cooling system. This cooling system must be capable of lowering the tool temperature to accelerate the sheet metal blank cooling rate

as well as sinking away the heat to the cooling fluid as fast as possible (Karbasian et al., 2010; Altan et al., 2012).

### 1.3 Objectives

To address the research problem stated in the previous section, the primary aim of this study would be to experimentally investigate the suitability and potential of nanocoolants to be introduced into the cooling channel of hot press forming (HPF) process. Thus, improving the mechanical properties of the hot pressed part and increasing the productivity of the automotive industry. The objectives of this research project are as follows:

- 1) To characterize the aluminium oxide,  $\text{Al}_2\text{O}_3$ /water-ethylene glycol mixture with optimum thermal properties by exploiting the properties of nanoparticles in terms of thermal conductivity and dynamic viscosity.
- 2) To evaluate the temperature distribution of the nanocoolants for quenching operation to replace chilled water in the HPF process by using experimental hat-shaped tools.
- 3) To analyse and compare the mechanical properties of boron steel product in terms of tensile strength and hardness with the microstructure transformation between the chilled water and nanocoolants.

### 1.4 Scope of Study

The scope of this research project is as follows:

- a) Nanocoolants with different volume concentrations of aluminium oxide,  $\text{Al}_2\text{O}_3$  nanoparticles from 0 to 1% are prepared by using the dilution process in a water-ethylene glycol mixture in this study due to the potential heat transfer rate in the engineering field and it is advanced fluids in the current trend.

- b) Several concentrations of nanoparticles are suspended in three different ratios by volume percent of water to ethylene glycol base fluids, which are 60%:40%, 50%:50% and 40%:60%, respectively. The mixture prevents the galvanic corrosion of the cooling channel system, and also prevent distilled water from boiling or freezing at extreme temperature.
- c) The thermal properties of nanocoolants are measured at a temperature range of 15 to 55 °C in a closed chamber. It is because the bulk temperature in the HPF process is 25 °C and the mean temperature of the hot formed tool is approximately 45 °C after the quenching operation.
- d) Boron steels are chosen as the sheet metal blank materials while SKD 61 is selected as hot formed tools in this research. It is due to that the boron steel can achieve ultra-high strength steel grade after austenitization process. SKD 61 defined as a hot work tool with higher contact heat transfer coefficient value.
- e) The thickness of the boron steel blank is 1.8 mm at the austenite temperature of 900 °C in 5 minutes heating time. The experimentally measured heat distribution is attained via *k*-type thermocouples located inside the hot forming tool. The selection of these austenitization parameters is explained in detail in Section 2.8.1 and 4.3.3.
- f) The analyses on the mechanical properties of the hot pressed parts are performed. The hardness and tensile strength test are conducted by using the Wilson Vickers 402 MVD machine and Universal Testing Machine model Instron 3369, respectively.
- g) Microstructure morphology evaluation cover from austenite transformation to martensite transformation phase for boron steel as delivered, chilled water pressed boron steel and nanocoolants pressed boron steel under the Light Optical Microscopy machine.



## 1.5 Hypothesis

Introduction of nanocoolants as cooling fluid in the hot press forming (HPF) process is anticipated to be an effective method to enhance the heat transfer coefficient value and the cooling rate during quenching operation. The higher the volume concentration of nanoparticles, the thermal conductivity and heat transfer value would be positively affected. The heat transfer distribution analysis of the nanocoolants and chilled water are mapped based on the experimental performance. At the end of the research, it is anticipated that the mechanical properties and microstructure transformation on the hot pressed part can be improved through the introduction of nanocoolants instead of chilled water. Thus, complementing of nanocoolants as an advanced heat transfer fluid to reduce the product cycle time in HPF process.

## 1.6 Thesis Organisation

The organisation of the thesis is arranged and discussed as follows, and the structure is shown in Figure 1.3.

Chapter 2 presents the review of the kinds of literatures starting from the background of nanocoolants and a comprehensive review on the technology of high strength steel (HSS) materials as well as the most recent research for the hot press forming (HPF) process.

The information on the raw nanocoolant materials, the procedure of preparation, measurements, and investigations on the thermal properties of nanocoolants are covered in Chapter 3. This chapter demonstrates the experimental investigation of thermal conductivity and dynamic viscosity of aluminium oxides,  $\text{Al}_2\text{O}_3$  suspended in water-ethylene glycol mixture nanocoolant for the cooling channel of HPF tools application. Once the results from the characterization are obtained, the optimum convection heat transfer coefficient value is used to compare with chilled water.

Chapter 4 presents the heat transfer distribution analysis for the hot forming tools and heated blank. The temperature changes of the HPF tools and heated blank by

using nanocoolant and chilled water are compared with several periods of quenching time. This analysis is required for the improvement of HPF process in order to enhance the products mechanical properties and productivity of HPF process. The mechanical properties like tensile strength and hardness and microstructure of the hot pressed part was examined.

Chapter 5 summarises the thesis by drawing conclusion and contributions of the present study to the body of knowledge as well as suggestions for the future works.

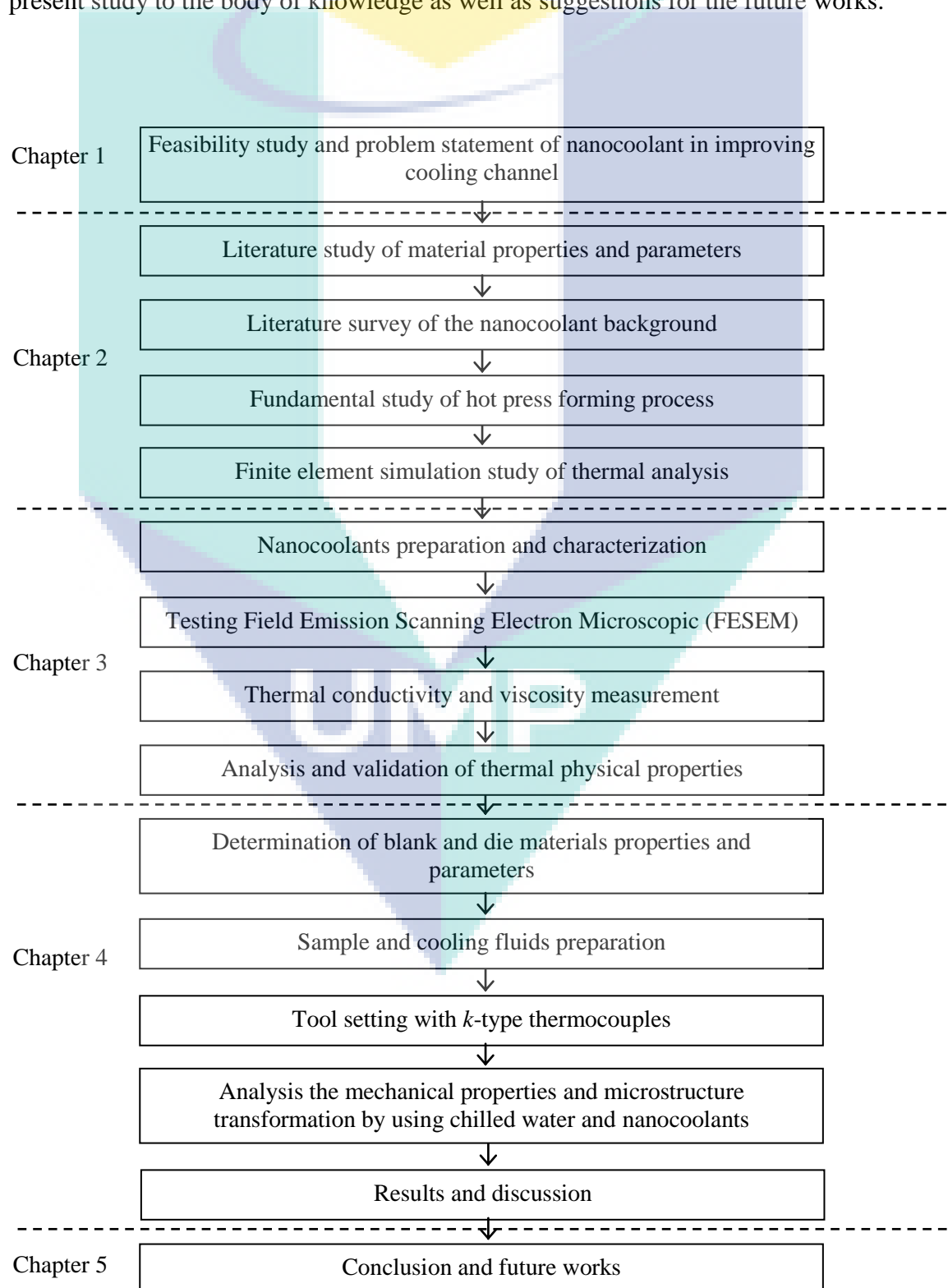
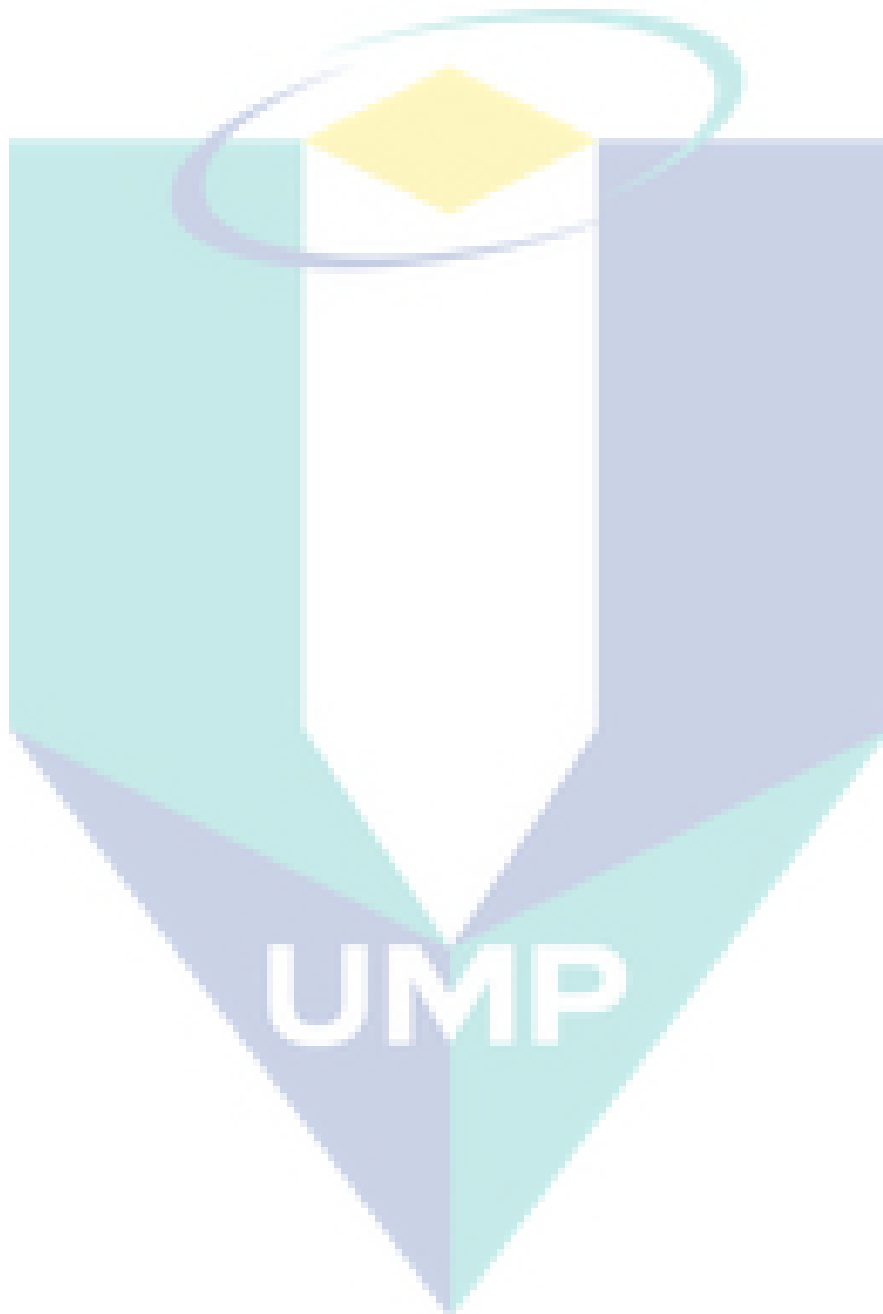




Figure 1.3 Thesis organisation



## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Introduction

In this chapter the significance of the general field of nanocoolants study and hot press forming (HPF) process have been reviewed from various relevant studies, and the research gap has been identified. This chapter provides a comprehensive review on previous researches related to the boron steel materials, HPF process and nanocoolants over the past decade. Topics such as the nanocoolants background, the preparation of nanocoolants, thermal properties, ethylene glycol as the fundamental in nanocoolants, boron steel and SKD 61 materials, heating method, transfer time, the heat transfer mechanism between tools and blank, and close up of previous studies on nanocoolants application were discussed. The significant findings in each study related to the dies and blank materials, heat transfer analysis and thermal physical properties such as thermal conductivity and dynamic viscosity are reviewed from various types of materials and base fluids. The review of nanocoolant studies that used ethylene glycol as the base fluid is provided to observe the current benchmark and research gap. The review structured chronologically from the background to the detailed study related to the present research as to give a clear understanding on the direction of the study.

#### 2.2 Background of Nanofluids as Coolant

Ultra-fine nanoparticles dispersed in a base fluid called nanofluid which is the new generation of heat transfer fluid and conventional cutting fluid. The base fluids can be engine oil, ethylene glycol, water or any lubricants. Krishna et al. (2013) stated that

augmentation of heat transfer and tribological properties of the nanofluids could serve as better cooling and lubrication agent during machining, hence making the production more viable. Masuda et al. (1993) investigated on the nanofluid to increase the efficiency of heat transfer equipment where the studies were concerned about thermal properties of the nanofluid. Nanofluid was first introduced by Choi et al. (1995) in the Argonne National Laboratory. The research experiments proved that nanofluids are superior heat transfer properties compared to conventional heat transfer fluids such as water and oil.

Heat transfer fluids are mainly used in the industrial and manufacturing company for cooling purposes. However, the conventional cutting fluids as a cooling medium in the industry, mostly exhibit poor performance regarding to thermal conductivity compared to solids (Krajnik et al., 2011; Amrita et al., 2013). Utilizing nanofluids as a cooling agent in machining is one of the novel concepts to replace the conventional heat transfer fluid. Due to their heat transfer and tribological properties, nanoparticles prepared from different materials such as aluminium oxide, zinc oxide, copper oxide and titanium oxide have been used in the machining operations. The applicability of nanofluids as coolant is considered because of the improved heat transfer coefficient of the fluid due to the solid particle inclusion (Eastman et al., 1997). Similar studies conducted by Rao et al. (2011a) to expand the nanofluid field with the investigation on many aspects related to the heat transfer performance of the nanofluids.

In the manufacturing industry, advanced heat transfer fluids with higher thermal conductivity and greater heat transfer coefficient are intensely needed. In recent years, the advances in nanoparticles synthesis techniques evidenced that the nanoparticles suspended in the base fluids can effectively substitute the conventional cutting fluids. Due to the nanofluids with engineered colloidal suspensions of nano-meter sized particles which are uniformly and stably dispersed in the base fluids attained higher thermal physical properties. There have been a lot of studies on heat transfer and the improvement of cooling capabilities are still in the developing stages in recent years (Krishna et al., 2013).

The value of thermal conductivity of different materials and conventional fluids has been summarized in Table 2.1. It can be seen that the thermal conductivity of solid

particles is much higher than the liquids. It is predicted that the thermal conductivity of fluids containing suspended solid particles could be significantly higher than that of the conventional fluids. However, the suspended micro-meter,  $\mu\text{m}$  or milli-meter,  $\text{mm}$  sized particles may cause some problems in the flow channels and rapid settling of particles in the fluid. Therefore, an innovative concept of nanofluid is the idea of using nano-meter sized particles to improve the quick sedimentation of particles in the fluid (Choi, 1998). Today, the diameters of particles range from the small size of a few nano-meters to hundred nano-meters and can be produced in a highly reliable process that can accurately control the particle size and shape.

Table 2.1 Thermo-physical property of different types of metal and liquid

Types of Metal	Thermal Conductivity, $k$ ( $\text{W m}^{-1}\text{K}^{-1}$ )	Reference(s)
$\text{Al}_2\text{O}_3$	36.0	Pak and Cho (1998)
ZnO	13.0	Bolz (2007)
$\text{TiO}_2$	8.4	Bontemps et al. (2008)
$\text{SiO}_2$	1.4	Vajjha et al. (2010a)
Types of Liquid	Thermal Conductivity, $k$ ( $\text{W m}^{-1}\text{K}^{-1}$ )	Reference(s)
Ethylene Glycol	0.258	Cengel (2011)
Engine oil	0.145	Cengel (2011)

### 2.3 Properties of Water-Ethylene Glycol Based Nanocoolants

In manufacturing field, it is prevalent to use water or conventional oil as a coolant in machining. However, the problem of applying water as coolant is that the water may either freeze or boil at extreme temperature conditions (Parker, 2003). Thus, some additives are added into the water to decrease the freezing point and to increase the boiling point of the water. The additives could avoid the coolant to freeze when the temperature is too low, on the other hand to reduce the system overheat when it is too hot. Ethylene glycol was the most common additive mixed with water. Ethylene glycol has the molar mass of  $62 \text{ g}\cdot\text{mol}^{-1}$  and chemical formula of  $\text{C}_2\text{H}_2\text{O}_2$ . According to the International Union of Pure and Applied Chemistry (IUPAC), ethylene glycol also named as ethane-1,2-diol. The solution is colourless, clear and syrupy texture liquid at room temperature. It is also soluble in water to form transparent solutions. In thermal engineering systems, ethylene glycol consists of hydroxyl and ethylene where the

hydroxyl part bonds with inorganic materials or water molecules. Hence, ethylene glycol can disperse well in the water (Hong et al, 2004).

Furthermore, the presence of ethylene glycol in extreme cold situation can avoid water to freeze at 0 °C temperature due to the lower freezing point of ethylene glycol which is -12 °C (CDC, 2003). While, the boiling point of ethylene glycol is much higher than that of water, which attains around 198 °C temperature (CDC, 2003). Figure 2.1 shows boiling point and freezing point of water and ethylene glycol mixture. It was shown that the mixture ratio of 60%:40% ethylene glycol/water did not freeze up to -45 °C (Peyghambarzadeh et al., 2011a). Hence, the solution with a high boiling point is required for the high temperature operating system to prevent damage to the system due to overheating.

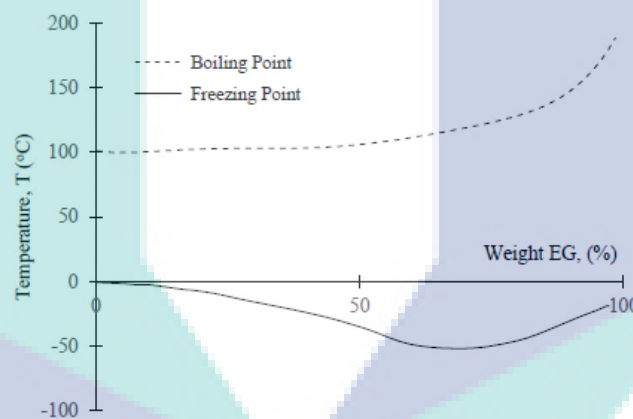


Figure 2.1 Freezing point and boiling point of water-ethylene glycol mixture  
Source: Peyghambarzadeh et al. (2011a)

Therefore, water and ethylene glycol mixture as a coolant for conventional thermal fluid is not a new concept in the thermal application. Coolant is used to cool down the system by flow through the instrument and prevent the instrument or system to be overheating. The coolant absorbed the heat produced by the device or system, and then transferred to the other places. A study from Denkena et al. (2014) stated that by controlling the volume flow rate of coolant can reduce the energy consumption of the machine in order to optimize the machine tools. These urged researchers in thermal fluids to use the mixture of ethylene glycol with high boiling temperature or low freezing point.

Recently, the most common choice of engine coolant has been a 50%:50% mixture of water and ethylene glycol in normal temperature and environment (Woodward and Gershun, 1993). For extreme cold temperature, the higher percentage of ethylene glycol that greater than 70% in the coolant mixture lead breakdown of inhibitors towards corrosion effect. This high percentage of ethylene glycol decreased the anti-freeze protection and reduced the heat transfer performance (Hull et al., 2008). In the year 2008, research on  $\text{SiO}_2$  nanoparticles suspended in 40%:60% of water-ethylene glycol mixture for three particle sizes of 20, 50 and 100 nm was reported (Kulkarni et al, 2008). Their experimental study on fluid dynamic characteristics and convective heat transfer enhancement with nanoparticle volume concentrations range from 2 to 10% with Reynolds number of 3000 to 12000. The research concluded that heat transfer coefficient increases as the increment of nanoparticle volume concentration and the effects are more significant for temperature around 0 °C. Bayat and Nikseresht (2012) conducted a research on forced convection heat transfer with water-ethylene glycol based  $\text{Al}_2\text{O}_3$  nanofluid at the temperature of 20 °C. The study was carried out with nanoparticles volume concentration of 1 to 10% and the Reynolds number from the range of 10000 to 100000. The thermal performance of nanofluids was measured by using  $K-T$  model, a dimensionless quantity model. The study showed that the convective heat transfer performance was enhanced with the increased of nanoparticle volume concentrations at the same Reynolds number. On the other hand, a significant pressure drop and pumping power were measured by using nanofluids in the turbulence region.

#### **2.4 Nanoparticles as Suspended Material**

Nanoparticles defined as ultrafine particles with the size below 100 nm which can be denoted as ten to the power of minus nine. Therefore, nanotechnology manipulates particles of nano-meter sizes that are much smaller than objects that obey Newton's laws of motion. However, it is not small enough as atomic level or simple molecule that is governed by quantum mechanics (Estelrich et al., 2014). Due to the development of modern nanotechnology manufacturing capabilities, varies the type of

nanoparticles that had been produced. These are evolving interests to the engineering research to explore the research area of nanoparticle for various potential applications, especially in microelectronic, automotive and medical fields.

Furthermore, one of the novel approaches is utilizing the nanoparticle in a base fluid as a cooling agent in the hot press forming (HPF) process to replace the conventional fluid cooling. The size of nanoparticles has a large surface area-to-volume ratio that influences the dispersible and performance of heat transfer between the nanoparticles and their adjacent environment. For instance, a 1 nm spherical particle has a surface area-to-volume ratio 1000 times greater than a 1  $\mu\text{m}$  particle (Singh, 2008). Therefore, the size of particles significantly affected the melting point and thermal properties. When the nanoparticles are in suspension form, the free mobility condition is significantly enhancing the heat transfer performance of the fluid. In an experiment, few types of nanoparticles in small amounts have been dispersed in a conventional solution, and the results showed the rise of thermal conductivity (Wu and Zhao, 2013).

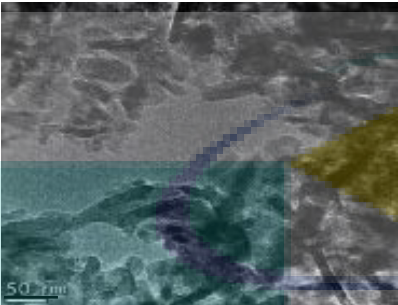
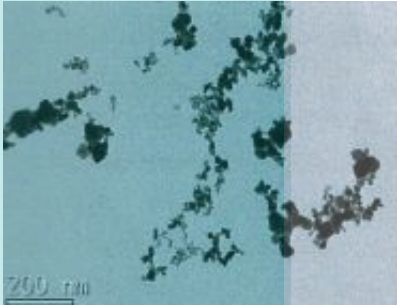
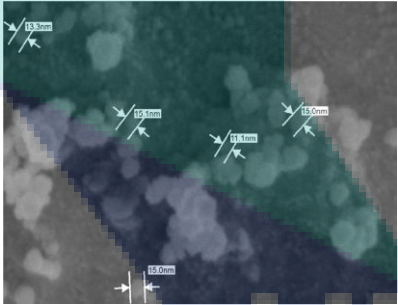
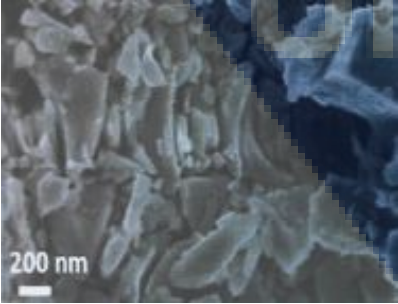
Physical properties of nanoparticles included their shape, size and morphological sub-structure. The actual shape and size of nanoparticles are identified through high magnification microscopes such as the Transmission Electron Microscopy (TEM) for particles suspended in liquid and Field Emission Scanning Electron Microscopy (FESEM) for solid particles. Lee et al. (2012) obtained TEM for nanofluid based  $\text{Al}_2\text{O}_3$  morphology determination. The  $\text{Al}_2\text{O}_3$  nanoparticles in nanofluid were observed to have the cylindrical shape and less than 50 nm size. Mariano et al. (2013) used TEM techniques to observe the structure and size distribution of ethylene glycol based  $\text{SnO}_2$  nanofluid in a study on rheological behaviour and thermal conductivity of nanofluids. The picture obtained from TEM showed that the composition of quasi-spherical shape and less than 100 nm size.

Abdolbaqi et al. (2016) had obtained the FESEM for water-bioglycol mixture based  $\text{Al}_2\text{O}_3$  nanofluids. The images revealed that the shape of the nanoparticles was spherical and size of the nanoparticles in nanofluid was measured to have an average diameter of 13 nm. In a study on rheological behaviour and thermal physical properties of SiC nanofluids, Nikkam et al. (2014) used the SEM machine to determine the morphology of the nanofluids. The picture showed that the hexagonal crystal structure



with an average nanoparticles size of approximately 115 nm. The summary of several types of nanoparticles and micrograph pictures are compiled in Table 2.2.

Table 2.2 Various types of nanoparticles and micrograph images

Micrograph Image	Types of Particles
	Author(s) : Lee et al. (2012) Micrograph : TEM Nanoparticles : Al <sub>2</sub> O <sub>3</sub> Shape : Cylindrical Size : 50 nm
	Author(s) : Mariano et al. (2013) Micrograph : TEM Nanoparticles : SnO <sub>2</sub> Shape : Quasi-spherical Size : <100 nm
	Author(s) : Abdolbaqi et al. (2016) Micrograph : FESEM Nanoparticles : Al <sub>2</sub> O <sub>3</sub> Shape : Spherical Size : 13 nm
	Author(s) : Nikkam et al. (2014) Micrograph : SEM Nanoparticles : α-SiC Shape : Crystalline Size : 115 nm



### 2.4.1 Nanoparticle materials

Several types of nanoparticles are produced by utilizing the advancements in the nanotechnology industry, but the selection of nanoparticles depends on the properties and application. Nanoparticles can be categorized into few types based on the material and structure of the particles. Many research investigations with different types of nanoparticles have been reported to enhance the thermal properties.

Pure metallic nanoparticles are the materials that have single ions such as silver (Ag), aluminium (Al), iron (Fe), copper (Cu), and gold (Au) (Monosson, 2013). The metallic nanoparticles are preferable for thermal application as they share the similar properties. Several studies of metallic nanoparticles suspended in base fluids have been carried out than the oxide nanoparticles. Liu et al. (2006) investigated that thermal conductivity of nanofluid based copper is enhanced by 23.85% compared to water when nanoparticles volume concentration of 0.1%. However, authors found that the thermal conductivity considerably decreased with the elapsed time when no surfactant was introduced in the nanofluids. Their chemical reaction with base fluid required to be considered for the application purpose.

Polymer and carbon nanotubes are majority used in the fundamental studies of nanofluids. The behaviour of multi-walled carbon nanotubes (MWCNT) mixed with water showed a significant increment of heat transfer efficiency compared to the pure water. Nevertheless, in a study of heat transfer of carbon nanotubes based nanofluid, they concluded that the enhancement of heat transfer depends on the condition of the flow and the volume concentration of carbon nanotubes with minimum pH effect (Ding et al., 2006).

Besides that pure metallic nanoparticles; polymer and carbon nanotubes, metal oxide nanoparticles such as carbide ceramics, oxide ceramics and nitride ceramics as ceramics nanoparticles are the first materials that are used to synthesize nanofluids (Hadadian et al., 2013). The reason behind the preferable is easy to prepare and its chemical stability. Each ceramics class developed distinctive material properties as they dispersed in the conventional heat transfer fluid. Among these kinds of ceramics, the most interested nanoparticles by the researchers are oxide ceramic nanoparticles. The

pioneer of the new class fluid such as Masuda et al. (1993) and Pak and Cho (1998) choose  $\text{Al}_2\text{O}_3$  nanoparticles with the unexpected finding of thermal conductivity enhancement to enhance heat transfer performance. This finding triggered interest for further development.

#### 2.4.2 Types of synthesized nanofluids

Nanofluids consist of two elements which are nanoparticles and base fluids. A variety of materials such as metal oxide, carbon nanotube, carbide, metal and hybrid are used in preparing the nanofluids. The selection of nanoparticles material depends on many reasons, including the application and performance. Initially, metal oxide and metal were used since the access to the material was easier where many manufacturers abundantly produced the materials.

In 2006, a group of researchers used copper nanoparticles suspended in an ethylene glycol base fluid by using a novel one-step method to prepare the nanofluids (Zhu et al., 2006). For thermal conductivity and heat transfer performance, Chun et al. (2011) used silicon carbide (SiC) with water base nanofluid to investigate the effect of nanofluid on a boiling thin platinum wire. Nikkam et al. (2014) used  $\alpha$ -SiC for investigation of rheological behaviour and thermal-physical properties. For a new modification of carbon nanotube (CNT), researchers such as Gao et al. (2012) and Esfe et al. (2014) for the study of nanofluid thermo-physical properties and heat transfer performance by using functionalized carbon nanotube (FCNT) and multi-walled carbon nanotube (MWCNT), respectively.

Another popular type of material used in the nanofluid field is the metal oxide such as copper oxide (CuO), silicon oxide ( $\text{SiO}_2$ ) and aluminium oxide ( $\text{Al}_2\text{O}_3$ ). Kulkarni et al (2008) investigated on the convective heat transfer enhancement with nanofluids consisting dispersion of  $\text{SiO}_2$  in the water-ethylene glycol mixture base fluid. For a study on the effective electrical conductivity of nanofluids, Granguly et al (2009) used  $\text{Al}_2\text{O}_3$  nanoparticles suspended in water. In 2014, researchers such as Azmi et al. (2014a), Said et al. (2014) and Hajjar et al. (2014) used several types of metal oxide nanoparticles in water dispersion such as  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$  and graphene oxide (GO). These studies were about the effects of temperature and nanoparticles volume

concentration on the thermo-physical properties and convective heat transfer performance of nanofluids under turbulent regions.

Currently, hybrid nanoparticles are the latest type of nanoparticles in nanofluids studies. More than two materials are combined to produce the hybrid material by using chemical processes. In 2011, Suresh et al. (2011) demonstrated an experiment on thermo-physical properties of nanofluids based  $\text{Al}_2\text{O}_3$ -Cu hybrid nanoparticles. Madhesh et al. (2014) used the hybrid nanoparticles of Cu- $\text{TiO}_2$  for investigation on rheological properties and heat transfer performance. The summary of several types of nanoparticles used in different studies is gathered in Table 2.3.

Table 2.3 Types of nanoparticles dispersed in nanofluids

Nanoparticles	Base fluid	Reference(s)
<i>Metal</i> Copper, Cu	EG	Zhu et al. (2006)
<i>Carbide</i> Silicon carbide, SiC $\alpha$ – type Silicon carbide, $\alpha$ -SiC	De-ionized water Water/EG (50:50)	Chun et al. (2011) Nikkam et al. (2014)
<i>Carbon Nanotube</i> Functionalized CNT, FCNT Multi-walled CNT, MWCNT	Water Water	Gao et al. (2012) Esfe et al. (2014)
<i>Metal Oxide</i> Silicon oxide, $\text{SiO}_2$ Aluminium oxide, $\text{Al}_2\text{O}_3$ Silicon oxide, $\text{SiO}_2$ Titanium oxide, $\text{TiO}_2$ Graphene oxide, GO Aluminium oxide, $\text{Al}_2\text{O}_3$ Titanium oxide, $\text{TiO}_2$	Water/EG (60:40) Water Water Water Water Water	Kulkarni et al. (2008) Ganguly et al. (2009) Azmi et al. (2014a) Hajjar et al. (2014) Said et al. (2014)
<i>Hybrid Particle</i> $\text{Al}_2\text{O}_3$ -Cu Cu- $\text{TiO}_2$	Water Water	Suresh et al. (2011) Madhesh et al. (2014)

## 2.5 Nanofluids Preparation

Nanofluids can be defined as synthesizing nanoparticles with base fluid where the nanoparticles remain in suspension for a certain time period. Two ways of well-known methods for preparation of nanofluids are two-step method and one-step

method. However, many studies used the two-step method in which nanoparticles are first produced as dry powders and then dispersed into the base fluid as the second processing step. The one-step method can be defined as a process of combining the nanofluid synthesis and preparation of nanoparticles directly in the heat transfer fluid. Figure 2.2 illustrates the technique of the one-step method for nanofluids synthesis.

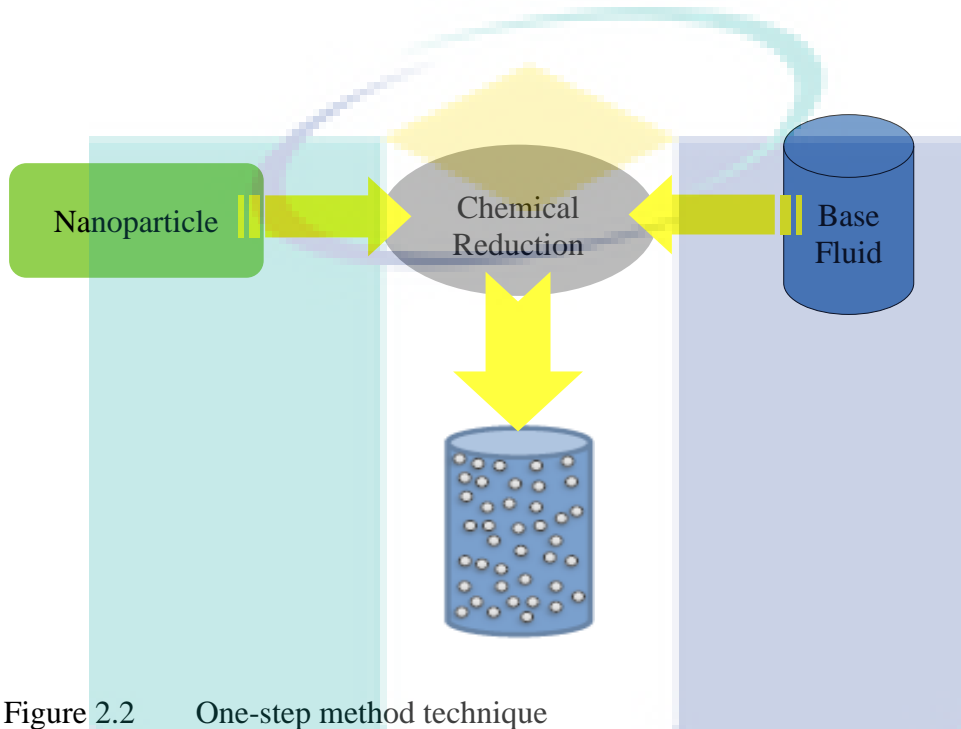


Figure 2.2 One-step method technique

Some researchers have used the one-step method to synthesize copper nanofluids (Eastman et al., 2001, Zhu et al., 2006, Liu et al., 2006). In one-step method, copper nanoparticles are contacted with ethylene glycol, and then under the nitrogen atmosphere by chemical reduction method to produce Cu-water nanofluids (Liu et al., 2006). This method was found to be an efficient and fast method that Cu nanofluids obtained better stabilization and higher reaction rate (Zhu et al., 2006). However, the disadvantages of the one-step method are very costly and the nanofluids cannot be produced on a large scale for industrial implementation. Regarding Yu and Xie (2012), nanofluids might have contaminated or impurity due to incomplete chemical reaction and preparation process in the one-step method.

Two-step method requires dispersion process for better stabilization of nanoparticle into a base fluid and is illustrated in Figure 2.3. According to Sidik et al. (2014), nanofluids preparation was using the two-step method begins by directly synthesizing nanoparticles with the base fluid. This method is the most extensively used

to process for preparing nanofluids (Suresh et al., 2011). Reddy et al. (2013) dispersed  $\text{TiO}_2$  with an average diameter of 20 nm size into ethylene glycol-water mixture as the base fluid. Lim et al. (2016) experimented  $\text{Al}_2\text{O}_3$ /water-ethylene glycol mixture nanofluid and found that nanofluid under ultra-sonication process for 1 hour has enhanced the stability of nanofluids for one month. The two-step method is the most cost-effective method to produce a large amount of nanofluids due to nanoparticle synthesis techniques have already been scaled up to industrial production level (Yu and Xie, 2012). Furthermore, ultra-sonication using an ultrasonic bath was used for nanofluids preparation to achieve uniform nanoparticles dispersion.

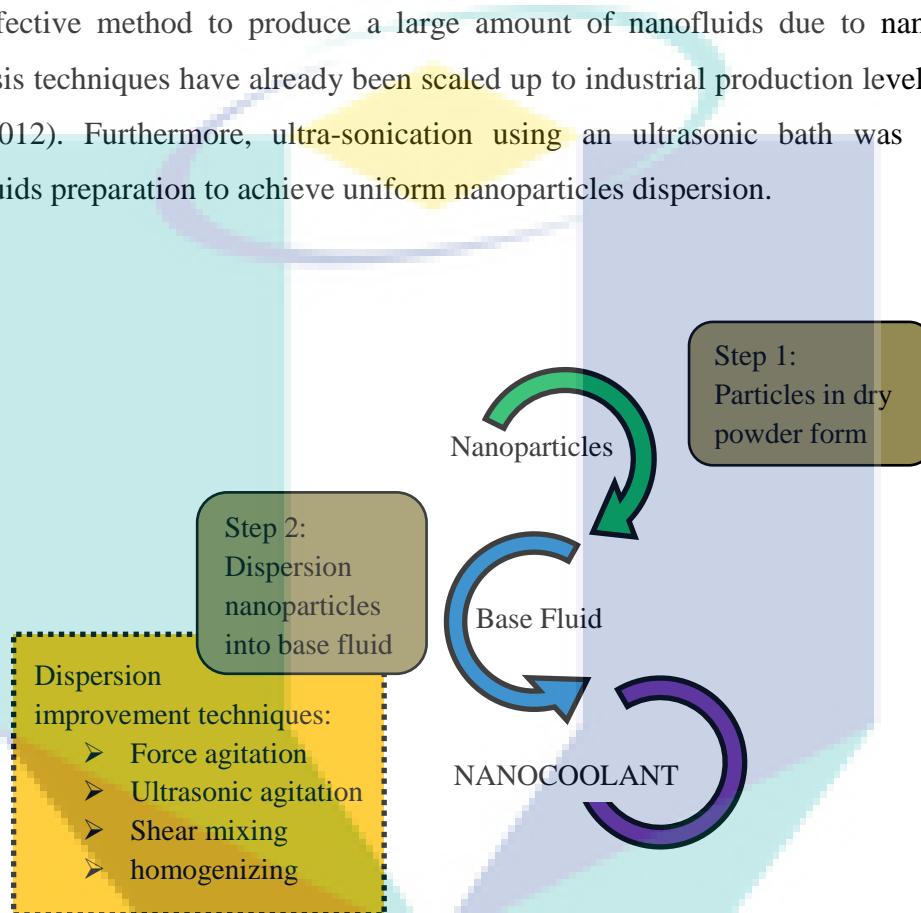


Figure 2.3 Two-step method technique

## 2.6 Thermal Properties of Nanofluids

Thermal properties of nanofluids include dynamic viscosity, thermal conductivity, density and specific heat. These thermal properties are related to the convection heat transfer coefficient,  $h$  and enhancement of heat transfer (Wen et al., 2009). The researchers found that the trend of heat transfer coefficient increases with effective thermal conductivity, density and specific heat but decreases with dynamic viscosity.

### 2.6.1 Dynamic viscosity

Property of collisions between particles and subsequent particles in a fluid that is moving at different velocity is named as dynamic viscosity. The graph of velocity pattern versus velocity is shown in Figure 2.4. Regarding the graph, shear stress is related to the velocity gradient when the fluid flows in the straight channel. The constant of proportionality is called as dynamic viscosity,  $\mu$  with the unit of  $\text{kg}\cdot\text{m}^{-1}\cdot\text{s}^{-1}$ , or  $\text{N}\cdot\text{s}\cdot\text{m}^{-2}$ , or Pa's (Cengel, 2010). However, the majority of researches suggested that the nanocoolants have the best performance with high thermal conductivity and low viscosity (Azmi et al., 2014a; Garg et al., 2008; Lim et al., 2016). Therefore, nanocoolants as heat transfer fluids are expected to show the increased of thermal conductivity without increased of pressure drop. Nanoparticles with base fluid mixtures are suitable to replace water because of the disadvantages of water such as high vapour pressure, high corrosive and limited operating temperature range (Timofeeva et al., 2011).

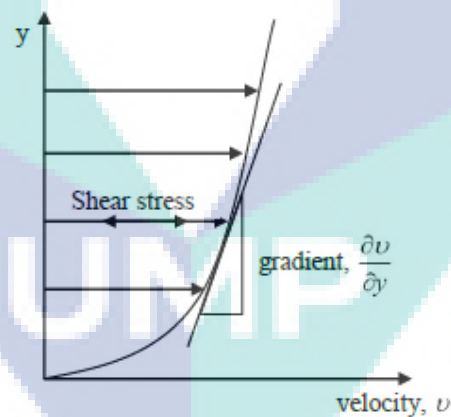


Figure 2.4 Graph of velocity pattern versus velocity

Source: Cengel (2010)

Furthermore, the dynamic viscosity of nanocoolants is not a constant variable which is affected by temperature difference and volume concentration (Yu et al., 2012; Vajravelu et al., 2013). The published research such as Namburu et al. (2007b) used CuO nanoparticles dispersed in 40%:60% water-ethylene glycol mixture and a temperature range from 10 to 50 °C. Authors found that the dynamic viscosity of nanocoolants decreased with the increase of temperature. The dynamic viscosity of

nanocoolant based CuO was enhanced about 73.21% compared with the base fluid. Another researcher, Sundar et al. (2012) observed that the dynamic viscosity of nanocoolants increased with the increment of volume concentration and decreased with the temperature range from 0 to 50 °C by dispersing Fe<sub>3</sub>O<sub>4</sub> nanoparticles in 80%:20% water-ethylene glycol mixture. The viscosity of Fe<sub>3</sub>O<sub>4</sub> at 1.0% volume concentration in 80%:20% mixture was enhanced by 2.94 times compared with the base fluid.

In 2013, one of the researchers conducted an experiment with 40%:60% water-ethylene glycol mixture based Al<sub>2</sub>O<sub>3</sub>. The results show that water-ethylene glycol based Al<sub>2</sub>O<sub>3</sub> nanocoolants present Newtonian behaviours for low concentration after the temperature below 40 °C (Said et al., 2013). Thus, nanocoolants viscosity not only depends on the temperature and volume concentration, but also the type of base fluid used to contribute to the nanocoolants viscosity. Moreover, Timofeeva et al. (2011) investigated the effect of base fluid and temperature to heat transfer performance. The experimental studies on SiC dispersed in water-ethylene glycol mixture of ratio 50%:50% showed that the efficiency of nanocoolants was higher for mixture based fluids compared to water based fluids. Also, the heat transfer performance of nanocoolants is enhanced by increasing in temperature as viscosity decreased. Table 2.4 shows the summary of dynamic viscosity investigations made by several researchers for different types of nanocoolants and their relevant findings.

Table 2.4 Dynamic viscosity investigations for different types of nanocoolants

Author(s)	Nanoparticles & Base Fluids	Temperature Range & Concentration Range	Findings
Namburu et al. (2007b)	CuO, W/EG (40:60)	T: -35 – 50 °C φ: 1.00 – 6.12%	Dynamic viscosity decreases with increasing temperature. ↓
Kole and Dey (2010)	Al <sub>2</sub> O <sub>3</sub> , W/PG (50:50)	T: 10 – 50 °C φ: 0.1 – 1.5%	Dynamic viscosity decreases with increasing temperature. ↓
Timofeeva et al. (2011)	SiC, W/EG (50:50)	T: 15 – 85 °C φ: 4.0%	Dynamic viscosity decreases with increasing temperature and mixture based fluids better than water based fluids. ↓
Kole and Dey (2011)	CuO, Gear oil	T: 10 – 80 °C φ: 0.5 – 2.5%	Non-Newtonian behaviour observed, as concentration increased.



Sundar et al. (2012)	Fe <sub>3</sub> O <sub>4</sub> , W/EG (80:20)	T: 0 – 50 °C φ: 0 – 1.0%	Dynamic viscosity enhanced by 2.94 times at 1.0% concentration. ↑
Yu et al. (2012)	Al <sub>2</sub> O <sub>3</sub> , W/EG (55:45)	T: 10 – 70 °C φ: 1.0 – 3.0%	Newtonian behaviour after temperature below 45 °C.
Said et al. (2013)	Al <sub>2</sub> O <sub>3</sub> , W/EG (40:60)	T: 25 – 80 °C φ: 0.05 – 1.0%	Newtonian behaviour in low concentration after temperature below 40 °C.

Table 2.5 Dynamic viscosity investigations for different types of nanocoolants (Continued)

Author(s)	Nanoparticles & Base Fluids	Temperature Range & Concentration Range	Findings
Jarahnejad et al. (2015)	TiO <sub>2</sub> , Water	T: 20 – 50 °C φ: 3.0 – 14.0%	Dynamic viscosity increased with increasing concentration, while it decreased with increasing temperature. ↑
Serebryakova et al. (2015)	Al <sub>2</sub> O <sub>3</sub> , W/EG (10:90)	T: 20 – 80 °C φ: 0 – 1.5%	Dynamic viscosity increased with increasing concentration. ↑

### 2.6.2 Thermal conductivity

Thermal conductivity is the property of a substance to conduct heat. The thermal conductivity of substances is temperature dependent. Nanoparticles disperse in conventional base fluids such as engine oil, water and ethylene glycol has been found to enhance the thermal performance compared to pure traditional base fluids (Choi, 1995; Azmi et al., 2013; Abbasi et al., 2014). Nanometer sized particles with a diameter of 1-100 nm suspended in base fluids are better than those fluids containing milli- or macro-sized particle regarding to particles sedimentation issue and significant enhancement of the effective thermal conductivity (Choi, 1995; Lim et al., 2016). The study of the thermal conductivity of a fluid plays vital role in the improvement of energy-efficient heat transfer equipment. Several experimental and theoretical investigations on increasing the thermal conductivity of water base fluids by suspending nanoparticles such as TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> have been conducted since the early 90's (Masuda et al., 1993). Currently, many researchers applied different types of nanoparticles to study the rheological behaviour of the nanofluids experimentally.



Sundar et al. (2013a) conducted a study on thermal properties using CuO with 27 nm sized particles suspended in 50%:50% mixture of water-ethylene glycol base fluid. They found that there was a positive augmentation of thermal conductivity of CuO nanofluid as the nanoparticles volume fraction and temperature of nanofluids increased. Lim et al. (2016) conducted an experimental investigation of thermal properties by dispersing Al<sub>2</sub>O<sub>3</sub> in a mixture of water-ethylene glycol base fluid and observed that the enhancement of thermal conductivity is about 10% higher than the base fluid in 1.0% concentration at 55 °C temperature. Apart from these two outstanding factors, thermal conductivity enhancement also found to be influenced by the types of base fluids and type of nanoparticles as well as the stability of the nanofluids (Chen et al., 2009; Pastoriza-Gallego et al., 2011; Javadi et al., 2013).

Ethylene-glycol based nanoparticles provided better heat transfer enhancement than water base nanofluids. It is proven by Maïga et al. (2004) in a study of heat transfer behaviour of water based  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and ethylene glycol based  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>. Another investigation of thermal conductivity found that thermal conductivity of nanofluids follows the behaviour of base fluid (ethylene glycol), where the maximum thermal conductivity was at roughly the same temperature in a pure base fluid (Beck et al., 2007). Researchers, Hwang et al. (2006) conducted thermal conductivity measurement of different types of nanoparticles such as copper oxide (CuO), silicon oxide (SiO) and multiwall carbon nanotubes (MWCNT) suspended in ethylene glycol and water base fluid. Each measurement showed enhancement of thermal conductivity as the different ratio of ethylene glycol base fluid. Table 2.6 concluded that the thermal conductivity study by several researchers using different types of nanofluids.

Table 2.6 Thermal conductivity study for diverse types of nanofluids

Author(s)	Nanoparticles & Base Fluids	Scope of Experiment	Enhancement of $k$ (%)
Lee et al. (1999)	CuO (35 nm) Ethylene glycol	Concentration: 4.0%	20
Beck et al. (2007)	Al <sub>2</sub> O <sub>3</sub> (20 nm) Ethylene Glycol	Concentration: 0 – 4% Temperature: 25 – 138 °C	↑
Timofeeva et al. (2007)	Al <sub>2</sub> O <sub>3</sub> (40 nm) Water Ethylene Glycol	Concentration: 0.5-10% Temperature: 10 – 60 °C	10 13
Yu et al. (2009)	ZnO (29 nm)	Concentration: 5.0%	26.5

	Ethylene glycol		
Jiang et al (2009)	CuO (29 nm) Al <sub>2</sub> O <sub>3</sub> (20 nm) R113	Concentration: 0.1 – 1.2%	3-7
Vajjha and Das (2010)	CuO (29 nm) ZnO (29 nm)	Concentration: 0 – 7% Temperature: 25 – 90 °C	18 21.4

Table 2.7 Thermal conductivity study for diverse types of nanofluids (Continued)

Author(s)	Nanoparticles & Base Fluids	Scope of Experiment	Enhancement of $k$ (%)
Parekh and Lee (2010)	Fe <sub>3</sub> O <sub>4</sub> (9.9 nm) Ethanol	Concentration: 4.7% Temperature: 25 – 65 °C	30
Pang et al (2012)	Al <sub>2</sub> O <sub>3</sub> (40 nm) SiO <sub>2</sub> (20 nm) Methanol	Concentration: 0.005 - 0.5% Temperature: 20 °C	10.74 14.29
Sundar et al. (2013a)	CuO (27 nm) Al <sub>2</sub> O <sub>3</sub> (36.5 nm) EG:W/50:50	Concentration: 0.2 – 0.8% Temperature: 20 – 60 °C	↑

Notation: ↑ - increased pattern

## 2.7 Sheet Metal of Boron Steel

Boron steel is one of the low carbon martensitic steel. Srithananan et al. (2016) stated that after the austenitization process, boron steel sheet metal only could achieve Ultra-High Strength Steel (UHSS) grade. The strength of boron steel is around 600 MPa as delivered form and the final formation of boron steel to obtain 1400 MPa strength after hot press forming (HPF) process (Naderi, 2007). The capability of boron steel to obtain complex geometry with good hot formability and optimum performance of mechanical properties are the primary purposes of using this material. Moreover, it is desirable owing to its exceptional fatigue strength and the absence of springback effect as hot formed boron steel (Dhillon, 2002).

Boron steel is categorised as low alloyed steel, and it is well-known for its ability to be strengthened through heat treatment to increase mechanical properties in terms of tensile strength and hardness. Boron steel contains several alloying elements such as Manganese (*Mn*), Silicon (*S*), Carbon (*C*), Titanium (*Ti*), Phosphorus (*P*),

Aluminium (*Al*), Sulphur (*S*) and Boron (*B*) as shown in Table 2.8. Typically, Boron is the most significant element that greatly improves the hardenability of boron steel sheet metal among the overall elements, while the existence of carbon determined the hardness of boron steel (López-Chipresa et al., 2008).

Table 2.8 Chemical compositions of boron steel weight percentage and mechanical properties before and after quenching operation

	Alloy Element							
	<i>Mn</i>	<i>Si</i>	<i>C</i>	<i>Ti</i>	<i>P</i>	<i>Al</i>	<i>S</i>	<i>B</i>
<b>22MnB5</b>	1.100 - 1.400	0.150 - 0.350	0.200 - 0.250	0.020 - 0.050	≤0.025	≥0.015	≤0.008	0.002 - 0.005
Mechanical Properties								
	Yield Strength (MPa)		Ultimate Strength (MPa)		Elongation (%)			
As delivered	400		600		25			
After quenching	1200		1400		5			

Source: Karbasian and Tekkaya (2010)

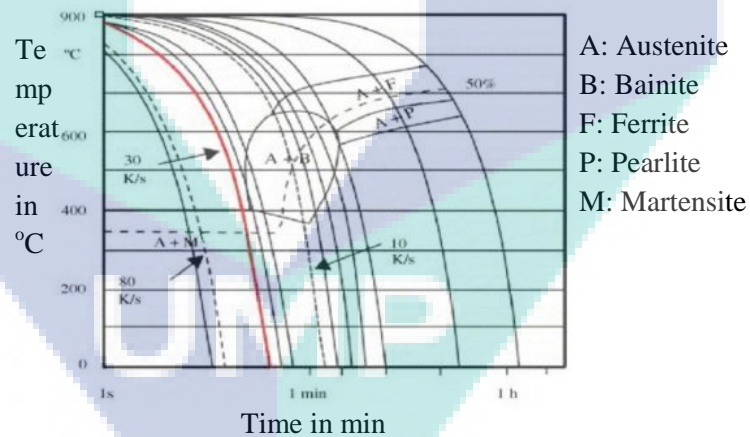


Figure 2.5 Temperature, time and transformation diagram of boron steel at various cooling rates

Source: Abdul-Hay et al. (2010)

Furthermore, annealed boron steel has a mixture of ferrite and pearlite phase microstructure with an ultimate strength of 600 MPa. The microstructure of boron steel gradually transformed into austenitic phase by heating about 830 °C and it is fully transformed at the temperature range of 900 to 950 °C (Karbasian and Tekkaya, 2010). When the austenitic phase of boron steel is quenched or immediately cooled down, its

phase is transformed to martensite or bainite phase or a mixture of both phases depending on its cooling rate as presented in Figure 2.5. In order to obtain the high strength of the final part with 1400 MPa, the austenitic phase must be fully transformed to the martensitic phase where it must be cool down at a rate of at least  $27 \text{ K}\cdot\text{s}^{-1}$  (Zhang et al., 2014).

The graph shows that the austenitic phase is continuously transformed into martensitic phase, which starts at the temperature of approximately  $400 \text{ }^\circ\text{C}$  during quenching operation and it is fully martensitic transformation at a temperature range of  $200$  to  $250 \text{ }^\circ\text{C}$ . In 2009, a researcher investigated metallurgical transformation of boron steel and found that HPF process significantly improves the mechanical properties of boron steel in terms of strength and hardness but reduces the percentage of elongation (Shapiro, 2009). It is because thermal properties of boron steel such as thermal conductivity,  $k$ , specific heat,  $C_p$  and elastic modulus,  $E$  are likely to vary with the change of material microstructure from the austenitic phase to martensitic phase. Table 2.9 concludes the summary of boron steel with different microstructure phases in temperature range from  $20$  to  $1000 \text{ }^\circ\text{C}$  during processing.

Table 2.9 Thermal-physical properties of boron steel

	Temperature, $T$ ( $^\circ\text{C}$ )										
	20	100	200	300	400	500	600	700	800	900	1000
$k$ ( $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ )	30.7	31.	30	27.5	21.7		23.6		25.6		27.6
$C_p$ ( $\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ )	444	487	520	544	561	573	581	586	590	596	603
$E$ (GPa)	212	207	199	193	166	158	150	142	134	126	118

Source: Shapiro (2009)

## 2.8 Hot Press Forming Process

In 1984, hot press forming (HPF) process was implemented in vehicle manufacturing to produce ultra-high strength steel (UHSS) boron steel. Currently, the HPF process exists in two different methods, namely direct and indirect process. The direct HPF process consists of a blank which is heated and fully austenite in a furnace for a certain period of time at temperature  $900 \text{ }^\circ\text{C}$ . Then, the heated blank is transferred to an enclosure forming tool by a transfer unit. However, the lower punch force also can produce any complex shape of parts due to sheet metal exhibits excellent ductility

properties at high temperature. (Lee et al., 2009). Finally, the heated blank is formed and quenched in a forming tool enclosure with coolant to produce the fully martensitic transformation part. Figure 2.6 shows the direct HPF process using a robotic arm to transfer the heated blank from the furnace to forming die.

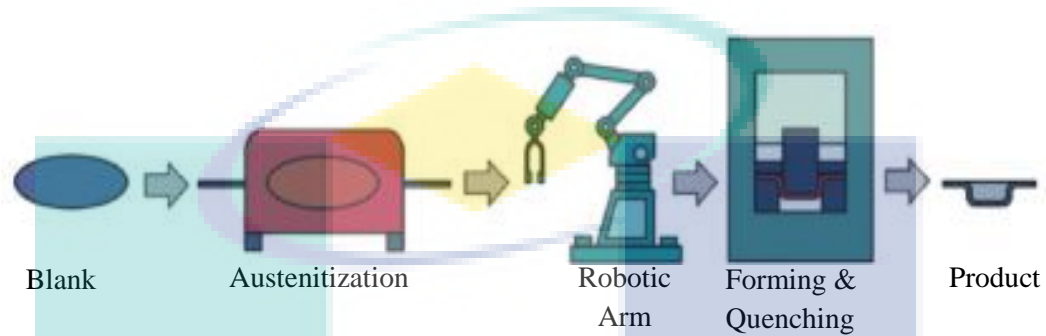


Figure 2.6 Direct hot forming process

Source: Karbasian and Tekkaya (2010)

Besides that, Figure 2.7 shows another HPF method namely as indirect HPF process which includes the conventional cold forming operation prior to the austenitization process. Indirect HPF process start from a part to be pre-formed, and drawn to approximately 90% of the final shaped in a cold die, then followed by a partial trimming operation (Hu et al., 2013). After that, it is heated in a furnace and quenched in the enclosure die. The reason for the additional step is to extend the forming limit for complex shapes by hot forming and quenching the pre-formed parts.

UMP

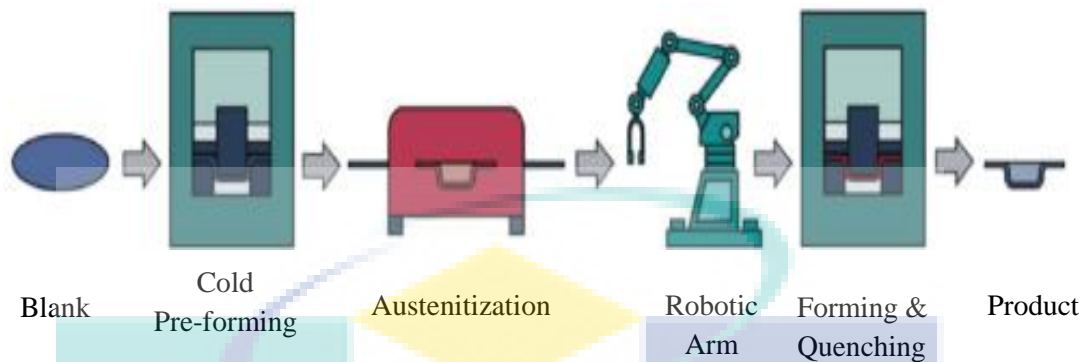


Figure 2.7 Indirect hot press forming

Source: Karbasian and Tekkaya (2010)

In 2008, the researchers stated that indirect HPF focused on quenching and calibration in the press after austenitization operation for complete cold pre-forming part (Merklein and Lechler, 2008). The difference between indirect hot press forming and the direct process is the pre-formed part is heated in the continuous furnace and quenched in the tools. Naganathan reported that pre-forming operation is added to extend the forming limit for the complex shapes by hot stamping and quenching of the cold pre-formed products (Naganathan, 2010). Product designs of indirect HPF begin with cold pre-forming dies design before heating up the blank in the furnace and HPF process. Lastly, the hot formed parts are applied in the automotive industry for chassis components such as bumper, A-pillar, B-pillar and roof rail (Hu et al., 2013).

Furthermore, sheet metal forming is crucial for cold pre-forming operation. The most general process is sheet metal bending which is used to form pieces such as hat-shape profile. It is also to enhance the stiffness of sheet metal by increasing its moment of inertia (Hu et al., 2002). Sheet metal with the hat-shaped profile is obtained when two parallel bending axes are produced in the same bending process and then a backing pad is used to force the sheet contacting with the punch bottom (Boljanovic et al., 2006; Bar-Meir, 2009). Figure 2.8 shows the bending process to produce hat-shaped workpiece. In a hat-shaped bending process, the bending line (B) is present at two locations and the bending force with the double value of bending line length is applied to sheet metal (Misumi, 2015). In this project, hat-shaped profile bending as a sheet

metal blank in HPF process for introducing nanocoolants of improvement cooling channel performance.

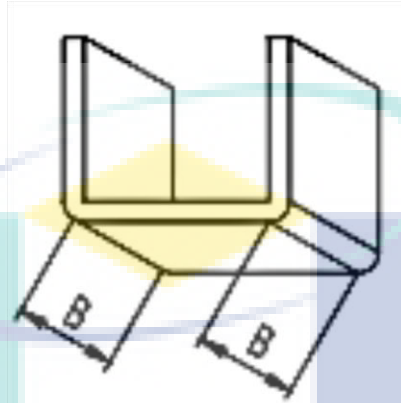


Figure 2.8 Hat-shape profile bending operation

Source: Misumi (2015)

### 2.8.1 Heating operation

The hot press forming (HPF) process starts with heating of the blank up to the austenitization temperature. In 2008, Lechler and his team demonstrated the annealing test by considering austenitization temperature and the time period for the determination of a homogeneous austenitization of the quenchable sheet metal 22MnB5 during HPF as a pre-condition for the desired fully martensitic transformation. In the experiment, the blanks were quenched through a full metallic contact pressure of 40 MPa on both sides. To evaluate the occurring phase transformation, the hardness of the quenched blanks was measured according to the standard of Vickers HV10. The fully martensitic microstructure and maximum hardness of approximately 470 HV of the quenchable sheet metal 22MnB5 were achieved with the austenitization duration time of 3 to 5 min from 900 to 950 °C austenitization temperatures (Karbasiyan and Tekkaya, 2010). The austenitization duration time increases with decreasing of furnace austenitization temperature.



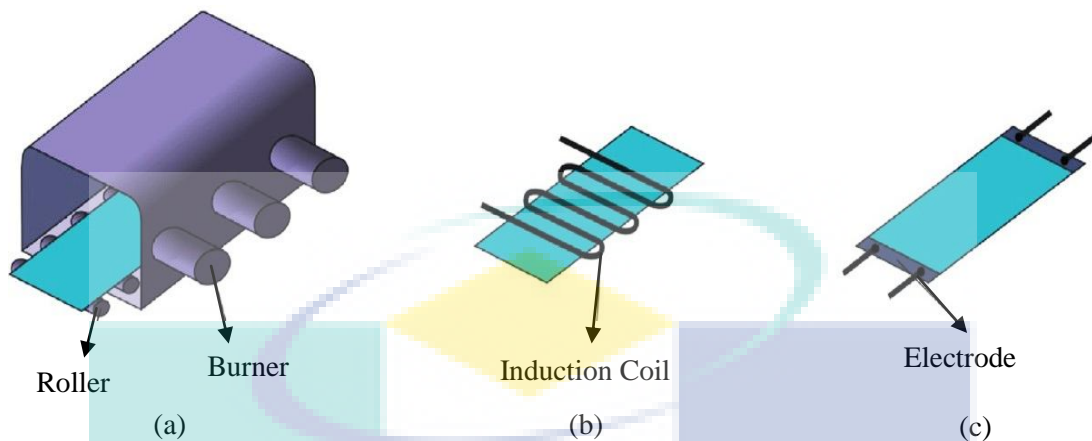


Figure 2.9 Various types of heating system (a) Roller hearth furnace, (b) Induction heating and (c) Electrical resistance heating

Source: Karbasian and Tekkaya (2010)

Thus, in the HPF process, the blank can be heated using three different thermal phenomena: induction heating, electrical resistance heating and radiation in a furnace as presented in Figure 2.9. The heating method would be different depending on the purposes either research or production. Radiation inside the furnace is usually practiced in the mass production industry and it is proven that capable of heating the sheet metal blank. While in the research area, the three heating methods could be applied depending on the adaptability and suitability of the operation to the experimental instruments (Karbasian and Tekkaya, 2010).

In the inductive heating operation, all electrically conducting or semiconducting materials can be heated by induction, and the resulting area of application is correspondingly large for this technology such as bulk forming, melting of metals and tempering. The geometry of the inductor determines the position of the magnetic field relative to the sheet metal blank, which causes different degrees of efficiency. The distance between blank and inductor also influences on the efficiency of the heating system. However, the electrical insulation between inductor and blank must be guaranteed, while shaped blanks tend to go out of shape when being heated (Karbasian and Tekkaya, 2010). An experiment conducted by Kollet et al. (2009) examined the capability of induction heating to heat up a blank to the austenitization temperature which was done by a special induction furnace. Figure 2.10a shows the two-step



induction heating elements and a conveyor system of the induction furnace. Figure 2.10b presents the result of induction heating with different heating power and conveyor speed. The energy efficiency of induction heating is up to two times higher compared to the roller heating furnaces or radiation inside furnaces, because of the higher losses of the roller heating furnace by exhaust gases and rollers.

The most ordinary type of furnace used in the radiation inside furnace method is the roller hearth furnace. The connected load and size of the furnace depends on the throughput and material to be heated. Then, the pre-sized blank is continuously moved into the furnace by a conveyor passing through the austenitization chamber where the radiation from the combustion of petroleum gas increases the temperature of blank (Karbasiyan and Tekkaya, 2010). The cycle time for the form-hardened part is mainly dependent on the dies closing time and the furnace residence time required to heat up the blank. However, the furnace with approximately 30 to 40 m length in hot press forming production line caused high space requirement. Compared with the method of radiation inside the furnace, electrical energy used to austenite the blank could reduce furnace residence time in order to decrease the cycle time.



UMP

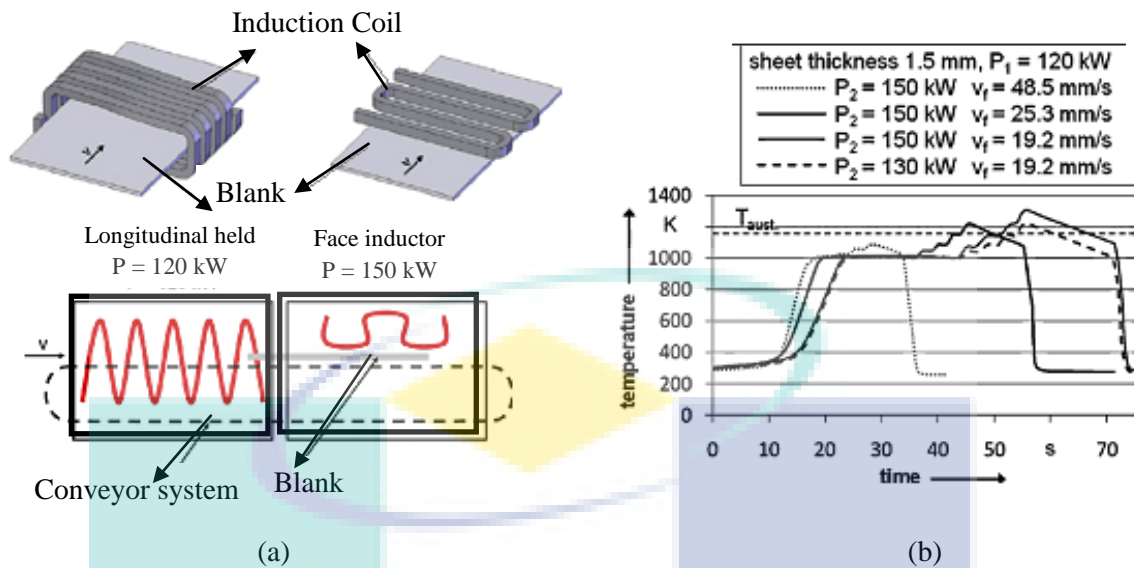


Figure 2.10 Induction heating operation (a) Schematics diagram of the customized induction furnace and (b) Variation of temperature curve as function of time for several feeding speed

Source: Kollect et al. (2009)

The principle of resistance heating is a blank clamped between the two pairs of electrodes as resistance for current passed through it (Mori et al., 2009). The resistance sheet metal caused the temperature of the part to heat up based on Joule's Law, which is due to that the heat generated in an electric circuit is proportional to the power of the electric circuit. Kollect and his team (2009) investigated heat transfer distribution on the sheet metal blank, and they discovered that a homogenous temperature distribution on blank with symmetrical shape could be achieved by properly arranging the position of the electrodes. Unfortunately, homogenous temperature or heat transfer distribution is very difficult for irregular and complex geometries blank shape due to the inconsistent distance between the negative and positive terminals (Behrens et al., 2008). Mori et al. (2012) conducted a study of resistance heating was suitable for localizing heating and spot heating, although this heating method has limitations on heating the wide surface area and irregular blank shape. An experiment was demonstrated to punch a small hole on a hardened boron steel sheet metal part, and the cutting operation was done at several temperatures ranging from room temperature to 800 °C.

Figure 2.11 shows a small area of the hardened boron steel part was heated up by using a pair of rectangular electrode integrated inside the cutting tools. According to the experimental results indicated that proper selection of input electrical energy is vital

for a resistance heating method to increase the temperature of sheet metal blank up to 800 °C in approximately 3 s. For this research project, the common heating furnace was used to austenite the sheet metal part inside the furnace. Then, the part was manually transferred to the HPF tools for quenching operation.

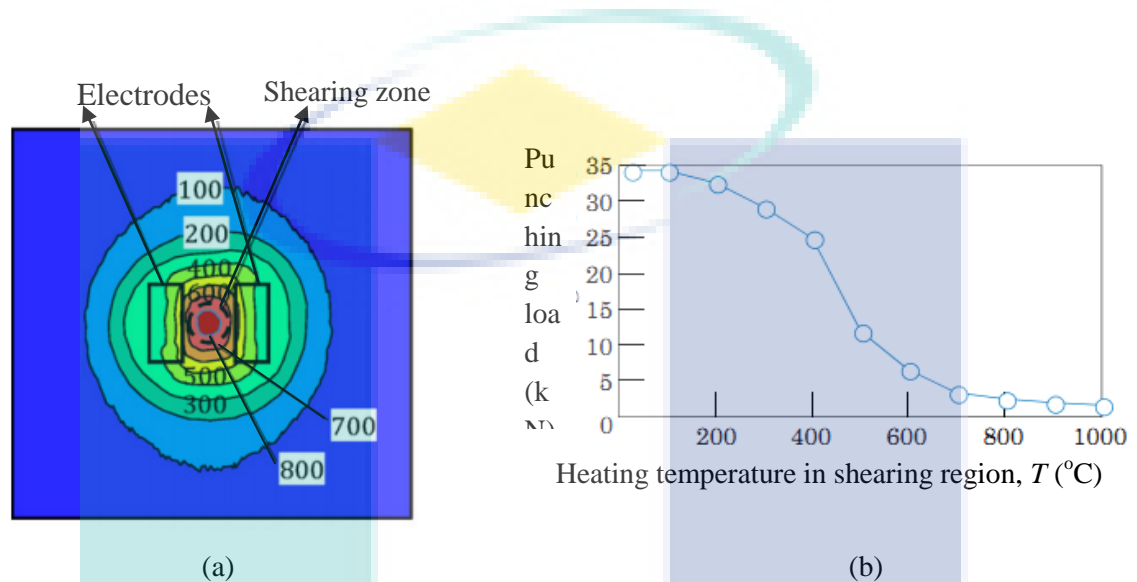


Figure 2.11 Localize heating (a) Positioning two rectangular electrodes on the blank and the temperature distribution diagram; and (b) Result of punching load for different heating temperatures in shearing region

Source: Mori et al. (2012)

## 2.8.2 Forming operation

Forming operation is a process where a sheet metal blank is drawn or plastically deformed into a specific shape of die and tool to produce the desired part (Banabic, 2010). Previously, the conventional cold forming operation was used to form sheet metal, but it was slowly replaced by hot press forming (HPF) process recently. Zhang et al. (2010) experimentally studied between hot press forming process and cold forming process, they found that the high dimensional accuracy can be obtained due to the low value of springback in HPF; and the thickness and weight of sheet metal can be reduced but the high strength part can be produced. Furthermore, the major parameter in a forming operation that needs to be considered are the behaviour of the materials and springback. The behaviour of material can be predicted by using the forming limit diagram (FLD) to simulate the result of material failure tests. Springback is when metal

tends to return to its original shape or contour after undergoing a forming operation (Kim et al., 2009).

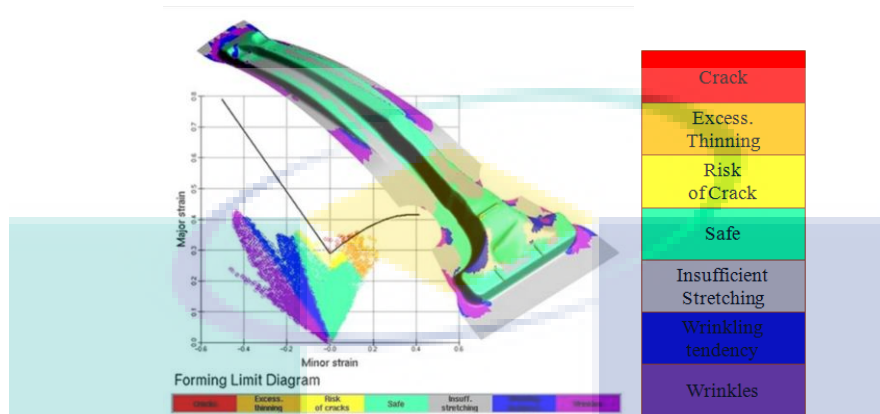


Figure 2.12 Forming Limit Diagram of B-pillar

Source: Skrikerud et al. (2010)

The forming limit diagram is used for forming the sheet metal to predict the forming behaviour of the blank (Cui et al., 2015). The diagram demonstrated graphical description of the material failure tests in order to determine the failure zone and the mechanical test is performed as shown in Figure 2.12. According to the simulation of B-pillar for geometry and tools, the initial temperature of sheet metal blank is 800 °C and tool surface temperature is approximately 200 °C. Figure 2.13 attempts to provide a simulated graphical description of the sheet metal part failure if it was formed at room temperature.

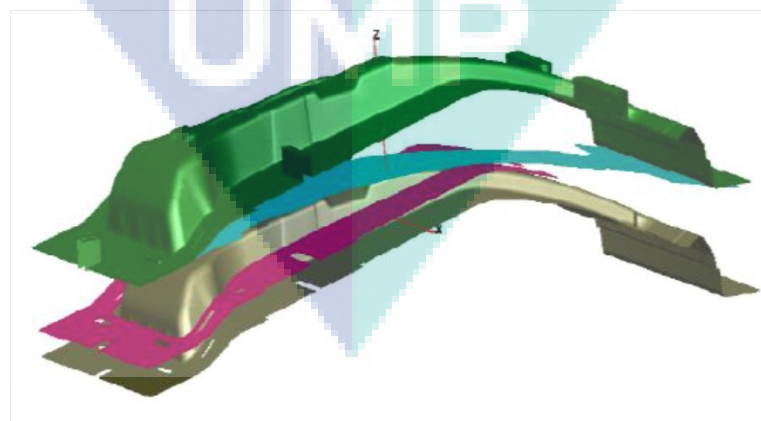


Figure 2.13 Simulation of B-pillar in hot press forming process

Source: Skrikerud et al. (2010)

Moreover, an experiment on the springback of high strength steel (HSS) after warm and hot sheet metal forming was performed by Yanagimoto and his crews (2005). They concluded that the springback of the hot steel sheet is reduced when the temperature of the steel sheet is beyond the recrystallisation temperature of austenite. They also cautiously changed the temperature of steel sheet for further investigation. Figure 2.14 shows the two different positions of A and B are selected for the plastically deforming region and flange zone, respectively.

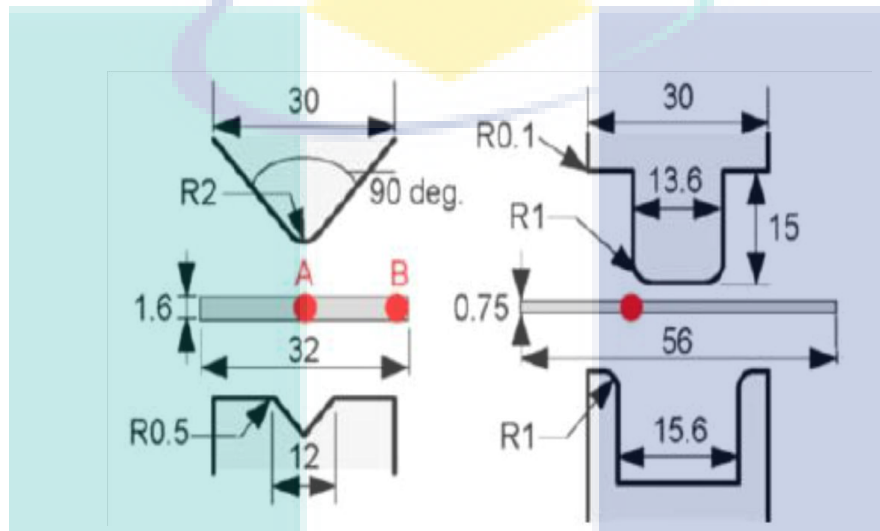


Figure 2.14 Geometries of tools and steel sheet for hat-shape and V-shape  
Source: Yanagimoto et al. (2005)

Regarding to the experimental study of Yanagimoto et al. (2005), springback effect may be reduced when the temperature of a plastically deformation region is less than the recrystallisation temperature of austenite. Figure 2.15 presents the results of springback decreases with the temperature at bending zone higher than 750 K but the temperature in flange zone is higher than 1000 K. Therefore, springback can be significantly reduced when the temperature of the bending area was at least in the warm forming range of 750 K and above.

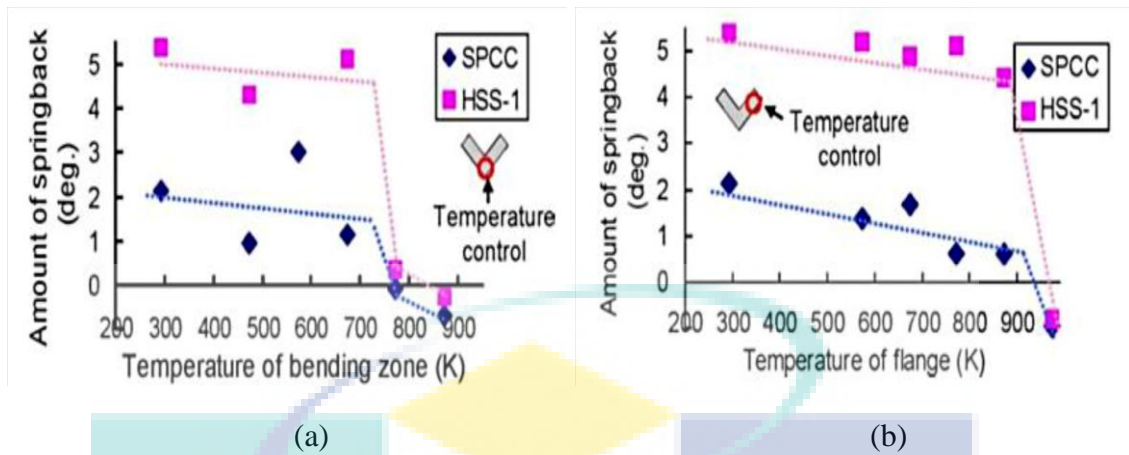


Figure 2.15 Degree of springback after V-shape bending with the relationship of temperature in (a) Bending region; and (b) Flange region

Source: Yanagimoto et al. (2005)

### 2.8.3 Punching and loading force

Ultra-high strength steel (UHSS) sheets have excellent strength, a hot forming operation with resistance heating was developed to reduce the punching force of UHSS sheet metal (Mori et al., 2005). The punching force or shearing load of UHSS sheet was decreased to improve the tool and dies life by heating the sheet. Mori et al. (2005) conducted a research of SPFC 980 sheet (UHSS) with a thickness of 2.0 mm was formed at elevated temperatures. Resistance heating was implemented in this research investigation which prevented oxidation and temperature decrement of the blank during warm and hot forming. The dimension of SPFC980 sheet metal is 130 mm length and 50 mm width, only 5 mm of each edge of the length is in contact with the electrodes as shown in Figure 2.16. The punch die with 10 mm in diameter was made of SKH 51 and coated with TiCN. A stamping force of 8 MPa was applied with the sheet metal holder to acquire sufficient contact between the blank and electrodes for electrifying, which the current flow through the blank as resistance. Table 2.10 shows the different temperature conditions of hot forming operation.

According to the experimental results of Mori et al. (2008), the increment of sheet metal austenite temperature, the smaller the stamping force to be given in the UHSS sheet. Figure 2.17 displays the relationship between the punching load and

heating temperature of UHSS sheet. Based on the graph, the stamping force increased when the heating temperature was below 650 °C. Therefore, the forming operation of UHSS sheet metal is effective for warm and hot temperature conditions. Besides that, the advantages of austenite temperature are the decrease in shearing or punching load and also the improvement in the sheared edge quality (Mori et al., 2008).

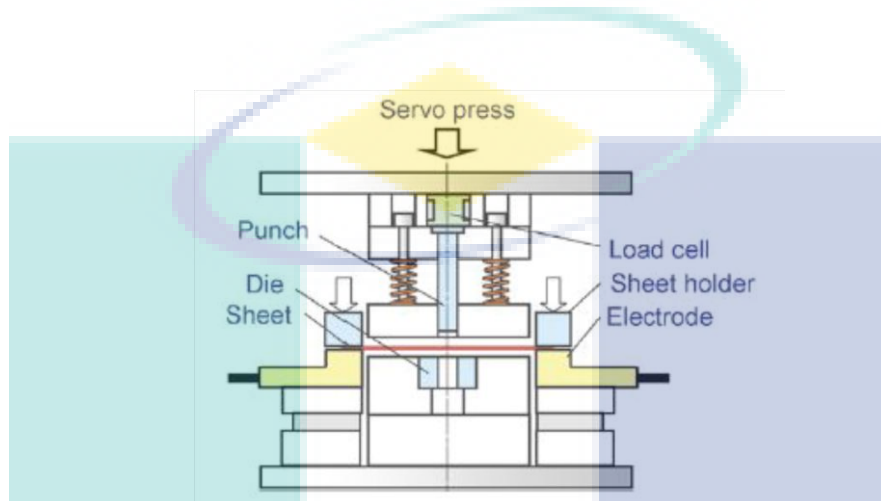


Figure 2.16 Hot forming process of UHSS sheet metal by using resistance heating  
Source: Mori et al. (2008)

Table 2.10 Stamping process in hot temperature conditions

Parameters	Number
Austenite Temperature, $T$ (°C)	650, 700, 830, 970, 1070
Stamping Velocity ( $\text{mm}\cdot\text{s}^{-1}$ )	150
Clearance Ratio, $c$ (%)	5, 10, 15

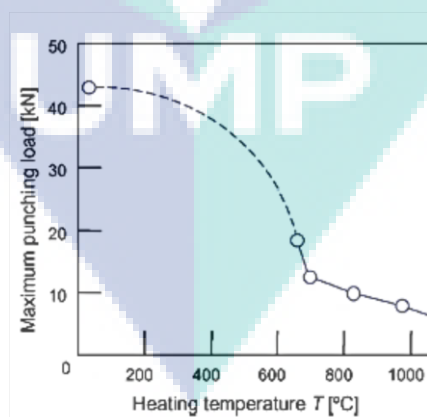


Figure 2.17 Relationship between the maximum stamping load and heating temperature of UHSS sheet metal

Source: Mori et al. (2008)



## 2.9 Heat Transfer Mechanism

Heat is defined as a form of energy that can be transferred from one system to another as a result of temperature difference (Abdul-Hay et al., 2010). A thermodynamic analysis is concerned with the amount of heat transfer as the system experiences a process from one equilibrium to another. Heat transfer is the determination of the rate of energy transfer. Hence, the transfer of energy as heat is always from the higher temperature medium to the lower temperature medium, and heat transfer equilibrium when the two mediums attain the same temperature (Cengel and Ghajar, 2011).

However, the thermal contact resistance is an interface which provides some resistance to heat transfer and it is per unit interface area. When two such surfaces are through against each other, the peaks form good material contact but the valleys form voids filled with air. Thus, an interface contains numerous air gaps of varying sizes which act as insulation because of the low thermal conductivity of air (Cengel and Ghajar, 2011). There are several factors that lead to the factor dependency of heat transfer which are the surface condition, fluid flow types, tools temperature and contact pressure.

### 2.9.1 Surface condition

Surface condition can be identified as the perfect contact exists at the interface of two layers and no temperature drop occurs at the interface zone from the analysis of heat conduction through multilayer solids. This would be the case when the surfaces are perfectly smooth, and a perfect contact at each point is obtained (Cengel, 2006). Figure 2.18 illustrates the temperature distribution and heat flow lines along the two solid plates pressed against each other in the case of perfect and imperfect thermal contact.



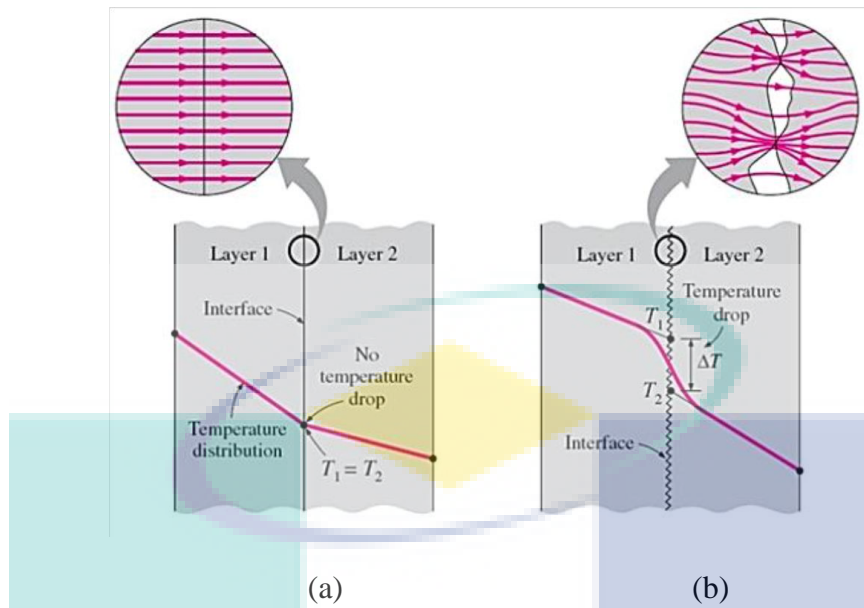


Figure 2.18 Thermal contact resistance (a) Ideal thermal contact; (b) Actual thermal contact

Source: Cengel and Ghajal, (2011)

### 2.9.2 Fluid flow types

Fluid flow can be classified as either laminar flow or turbulent flow. Laminar flow is defined as the highly organized fluid motion and it is characterized by smooth layers of fluid. Moreover, turbulent flow is the highly disordered fluid motion that typically occurs at high velocity and it is characterized by velocity fluctuations. While, the factor that determined the type of fluid flow is the ratio of inertia forces to viscous forces within the fluid and it is expressed by the non-dimensional Reynolds Number,  $Re$  which is given by the Equation 2.1 (Holman, 2009).

$$Re = \frac{\rho v D}{\mu} \quad (2.1)$$

where,  $\rho$  is the density of the fluid;  $v$  is the fluid velocity;  $D$  is a fluid characteristic distance; and  $\mu$  is the viscosity of thermo-physical properties. As an example of fluid flow in a pipe,  $D$  will be the pipe diameter and  $v$  will be the average fluid velocity. Typically, viscous stresses within a fluid tend to stabilize and organize the flow,

whereas excessive fluid inertia tends to disrupt the organized fluid flow and lead to disordered turbulent behaviour (Cengel and Ghajar, 2011).

Fluid flow can be determined as a laminar behaviour for Reynolds Numbers not more than 2000 values. However, fluid flow is completely turbulent behaviour when the Reynolds Number is beyond 4000 values. When the Reynold Numbers is in between 2000 and 4000, the fluid flow is neither laminar nor turbulent but it is in transition behaviour, and it is possible to find sub-regions of both flow types within a given flow field (Incropera et al., 2002). Cengel and Ghajar (2011) stated that highest heat convection happened in turbulent fluid flow and better convection heat coefficient would contribute to the increase the cooling rate in order to enhance the cooling system. Newton's law of cooling expresses that the rate of heat transfer from a surface at temperature  $T_s$  and transfers into the surrounding fluid at temperature  $T_f$  is given by the Equation 2.2.

$$\dot{Q}_{convection} = hA(T_f - T_s) \quad (2.2)$$

where the heat transfer coefficient,  $h$  has the units of  $\text{Btu}\cdot\text{s}^{-1}\cdot\text{in}^{-2}\cdot\text{F}^{-1}$  or  $\text{W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$ ; and  $A$  is the area of substances. While,  $T_f$  and  $T_s$  are ambient temperature and surface temperature, respectively. This equation is a simplified correlation between the fluid state and flow conditions. Hence, it is typically named as a flow property but the heat transfer coefficient is not a thermodynamic property (Cengel and Ghajar, 2011). In this project, the equation of convection heat transfer above is used to simulate the thermal analysis in turbulent flow behaviour, which is applied for highest heat convection.

### 2.9.3 Tools temperature and contact pressure

Heat transfer coefficient (HTC) at the blank-dies interface has been studied and investigated under a certain value of pressure by Bosetti et al. (2010). He and his company demonstrated an experiment to identify HTC in hot stamping of boron steel sheets under the condition which was very close to the industry. In the experiment, the process involved the compression of the sheet metal blank between two flat tools in

imposing value of applied pressure without large scale sheet deformation. The quenching tools consisted of two 10 mm thick exchangeable water cooled contact plates and thermocouple equipped specimen as shown in Figure 2.19. The specimen was undergoing a previous homogeneous austenitization at a temperature of 900 °C in a furnace and manually transferred on four springs seated pins under atmospheric conditions. Similar experiments were also conducted by Merklein and Lechler (2008) by using a testing software to measure the specimen loaded during quenching with a defined nominal contact pressure up to 40 MPa. The temperature values of both contact plates and sheet metal blank were gathered by using integrated Ni/Cr-Ni-thermocouples during the experiment.

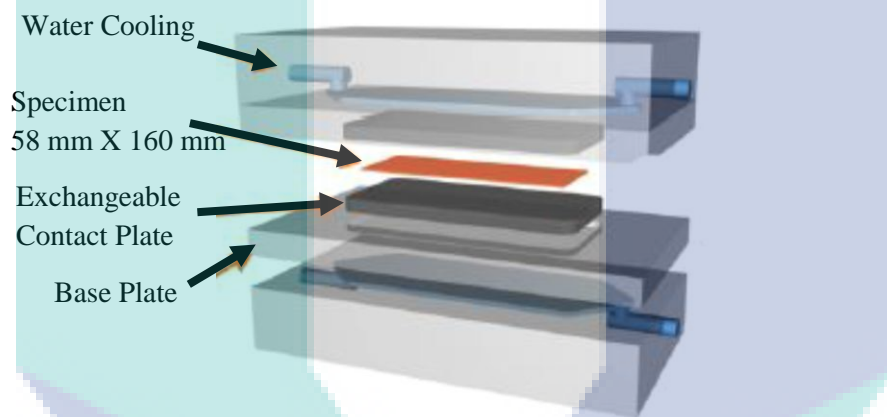


Figure 2.19 Experimental setup and instrument for heat transfer coefficient testing

Table 2.11 Results of contact pressure and standard deviation of HTC

Contact Pressure (MPa)	HTC ( $\text{W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$ )	Standard Deviation ( $\text{W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$ )
0	1231	82
5	1484	27
10	2025	37
20	2395	69
30	3065	117
40	3308	159

Source: Bosetti et al. (2010)

Table 2.11 presents that the value of heat transfer coefficient increases with an increment of contact pressure and at the same time the standard deviation was also increased. Contact pressure is directly proportional to the heat transfer coefficient. Merklein et al. (2006) stated that the tool temperature is also directly proportional to the

heat transfer coefficient. Figure 2.20 shows that the heat transfer coefficient increases as the temperature of tools increases.

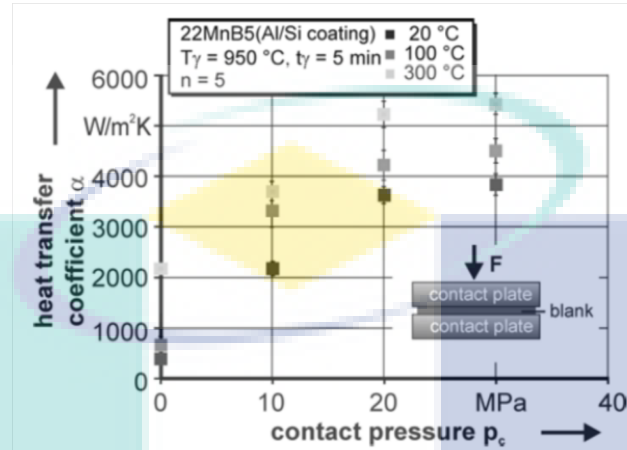


Figure 2.20 Relation between HTC as function of contact pressure for different tool temperatures

Source: Merklein et al. (2006)

## 2.10 Cooling Channel System of Die Design

One of the vital equipment in hot press forming (HPF) production is the hot forming die. The crucial factors that need to be considered in the die design are the manufacturing cost of hot forming, productivity and quality (Hu et al., 2013). Moreover, die material, die clearance, cooling channel design and effect of heat transfer must also be considered in the die design concept development (Hoffmann et al., 2007). Besides that, it must also achieve the HPF process system requirement in terms of austenitization temperature transmitting time and the rate controlling for hot forming. However, some issues such as wrinkling, cracking, no uniform thickness distribution and failure to complete quenching were faced in dealing with ultra-high strength steel (UHSS) blank in HPF (Naganathan and Penter, 2012).

Hot forming tools used in HPF process are similar to typical stamping process except for the additional cooling system that is machined inside the tool. The objective of the cooling channel design is to quench or rapidly cooling down the hot formed part effectively and to achieve a cooling rate of at least  $27 \text{ K}\cdot\text{s}^{-1}$  to attain martensitic

transformation (Karbasiyan and Tekkaya, 2010). The main part of the overall tools for dies and instance punch are required to be designed to cool down sufficiently to ensure the excellent characteristics of the formed part (Merklein and Lechler, 2006). These main tools are connected to a chiller, which is a medium to cool and recycling the coolant. Figure 2.21 shows the HPF tool for forming process. It consists of an upper die and lower die that are used to form the sheet metal blank. The punch acts as a metal block that is used for forming the sheet metal blank. While, the blank holder is a part of the HPF die that holds the blank against the die to control the metal flow. Hoffmann et al. (2007) employed a similar experimental setup to determine the heat transfer coefficient from the HPF process as shown in Figure 2.22. In this project, the cooling channel design was preliminary studied and manufactured in the HPF dies for forming the hat-shaped part.

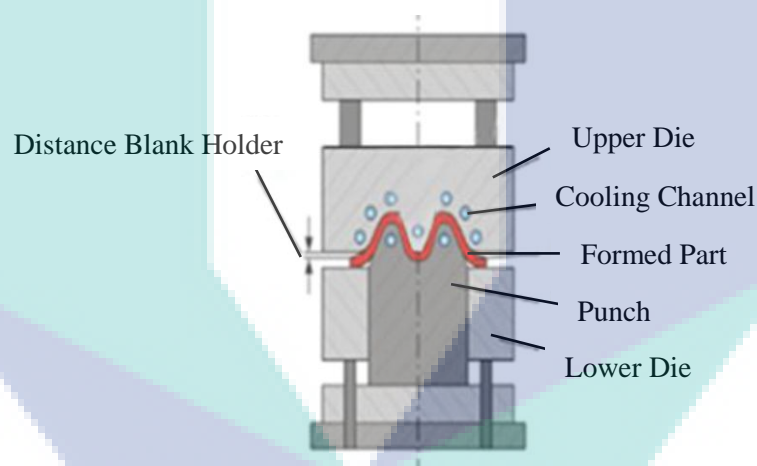


Figure 2.21 Schematic diagram of hot press forming tool design  
Source: Karbasiyan and Tekkaya (2010)

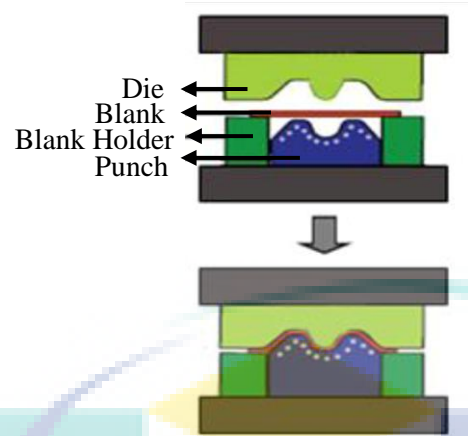


Figure 2.22 Schematic diagram of the quenching tool integrated with hot forming operation

Source: Hoffmann et al. (2007)

## 2.11 Summary

The studies of nanocoolants and hot press forming (HPF) process in this chapter have covered the thermal physical properties and experimental investigation on nanocoolant as the cooling medium for HPF tool application. Thermal conductivity and dynamic viscosity of nanocoolants behaved according to the particle size, temperature condition, volume concentration, types of materials and base fluids. The studies on dynamic viscosity and thermal conductivity of nanocoolants in a mixture of water-ethylene glycol are very limited. Only Kulkarni et al. (2008), Sundar et al. (2012), Azmi et al. (2014) and Said et al. (2014) conducted the experimental studies of  $\text{Fe}_3\text{O}_4$ ,  $\text{SiO}_2$  and  $\text{TiO}_2$  nanoparticles.

Following that the related previous studies using the nanocoolants base mixture are presented in this chapter. Most of the researchers found that the nanocoolants base mixture are stable for the convective heat transfer investigation and found that the significant enhancement in heat transfer performance. The enhancement of thermal conductivity using  $\text{Al}_2\text{O}_3$  base mixture nanocoolants is observed initially by Sundar et al. (2014). However, limited discussion on the influence of different ratio base fluids on the performance of nanocoolants are stated. Thus, the evaluation of thermal conductivity and dynamic viscosity for  $\text{Al}_2\text{O}_3$  nanocoolants were determined through

experimental measurement and characterization in Chapter 3 before obtaining the application in HPF process data.

In order to achieve a tool with high cooling efficiency as well as having homogeneous temperature distribution throughout the tool, the heat transfer of the tool must be in a perfect condition where, no temperature drops at the interface. HPF dies with the cooling channel can dissipate heat from the tool as well as to control the tool temperature. Cooling fluid is introduced inside the tool as the medium for cooling the tool. In addition, heat transfer coefficient can be determined by obtaining the equations involved Reynolds number and Prandtl number to produce the Nusselt number as reported by Lim et al. (2016). Due to the above parameters, the heat transfer experiment needs to be arranged to obtain the tool with high cooling efficiency as well as having homogeneous temperature distribution throughout the HPF dies. George et al. (2012) and Lin et al. (2015) evaluated the heat transfer distribution of the tool by placing the thermocouples approximately 4 to 5 mm away from the loading counter. The experiment results obtained from the present study would be reviewed and the trend would be compared for validation purposes. Namklang et al. (2016) validated their study by this method and acceptable agreement was found between experimental results.

However, the influence of the nanocoolant, water/EG mixture based  $Al_2O_3$  as a cooling medium in HPF process of heat transfer performance in forced convection heat transfer is still undiscovered. Hence the determination of heat transfer coefficient and cooling rate for the hot pressed part is shown in the subsequence Chapter 4. In Chapter 4, the microstructure transformation phase and the mechanical properties such ultimate tensile strength and hardness of the final product from the HPF process are examined. Naderi et al. (2011) and Lobbe et al. (2016) investigated the effect of different cooling rates and quenching time periods towards the tensile strength and hardness of the hot pressed part.



## CHAPTER 3

### NANOCOOLANT PREPARATION, CHARACTERISATION AND AUGMENTATION

#### 3.1 Introduction

In this chapter, the materials, solution and experimental procedures used in preparing the nanocoolants based aluminium oxide were briefly described. All the devices used to measure the thermal physical properties such as thermal conductivity and dynamic viscosity were explained item wise. The methods of finishing the nanocoolant experimental investigations of this project were presented briefly in Figure 3.1. Next, the data were collected from the stability of nanoparticles observation, thermal physical properties of thermal conductivity measurement and dynamic viscosity measurement; and convective heat transfer analysis in relation to this research objective. This research project initially studied a detailed stability evaluation through several observations such as scanning micrograph evaluations and sedimentation observation. This chapter also discussed the findings of thermal physical properties behaviour with respect to the concentration of nanoparticles and temperature. The discussion began with the validation of data with available literature, and proceeded with the nanocoolant data measurement and evaluation. The evaluation and measurement of thermal physical properties of nanocoolants were crucial as it was adapted into the investigation of convective heat transfer.

Moreover, data analysis of convective heat transfer involved different engineering parameters such as Reynold number, Nusselt number and heat transfer coefficient. The significant effect of the operating temperature to the overall performance of nanocoolant was subjected to the characterization of heat transfer coefficient and Nusselt number. This was the manner that the study included the effects



of increment of the operating temperature or bulk temperature, thus answering the research question and the usage of nanocoolant in real applications of nanocoolants in manufacturing cooling system. Figure 3.1 briefly presents the methods for completing the experimental investigation of thermal physical properties of nanocoolants in hot press forming (HPF) die application.

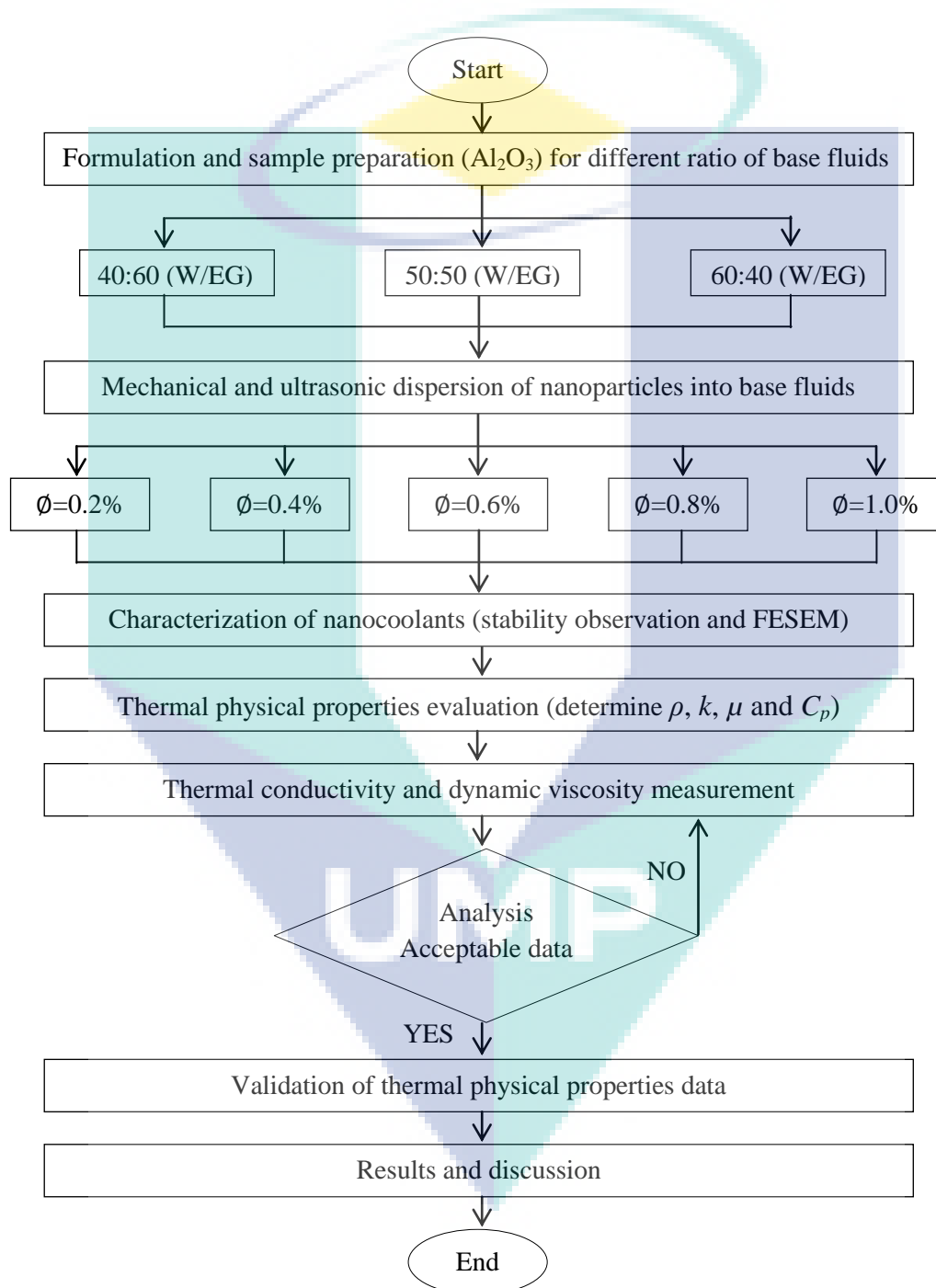


Figure 3.1 Flow chart of nanocoolant characterisation and experimental convection heat transfer

### 3.2 Materials and Preparation of Nanocoolants

Preparation of stable nanocoolants was the critical stage in engineering convection heat transfer experiment (Haddad et al., 2014). Sidik et al. (2014) conducted a review of the nanocoolant preparation and noted that the preparation of nanocoolant influenced their thermal conductivity. As described in Section 2.5, there are two well-known methods in the preparation of nanocoolants which are one-step and two-step techniques. The nanocoolants were prepared by dispersing the nanoparticles in different ratios of base fluid through proper mixing. After meticulous consideration, the selected nanoparticles was aluminium oxide ( $\text{Al}_2\text{O}_3$ ) with an average size of 13 nm and prepared by using the two step technique which was commonly used method for nanocoolant preparation (Azmi et al., 2013).

Nanocoolant was prepared by dispersing the nanoparticles in three different ratios of water to ethylene glycol base fluids, which were 60%:40%, 50%:50%, and 40%:60% respectively. The aluminium oxide ( $\text{Al}_2\text{O}_3$ ) nanoparticles in the powder form are used and procured from Sigma-Aldrich, USA. The fine particles of  $\text{Al}_2\text{O}_3$  have an average particle diameter of 13 nm with 99.8% purity as shown in Figure 3.2. The characterization of the  $\text{Al}_2\text{O}_3$  nanoparticle was obtained by Field Emission Scanning Electron Microscopy (FESEM) imaging technique. Figure 3.3 shows the image from FESEM model JSM-7800F with the magnification of X300,000.  $\text{Al}_2\text{O}_3$  nanoparticle shape is observed as spherical and the sizes are approximately 13 nm as shown in the FESEM image. The properties of  $\text{Al}_2\text{O}_3$  nanoparticles are shown in Table 3.1 (Sundar et al., 2014; Batchelor, 1977; Azmi et al., 2013; Lim et al., 2016).

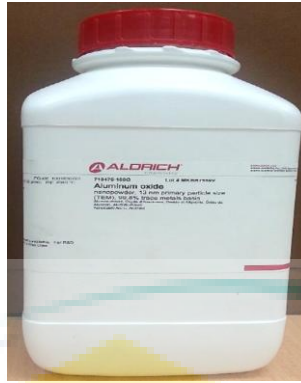


Figure 3.2 The Al<sub>2</sub>O<sub>3</sub> nanoparticles with size of 13 nm

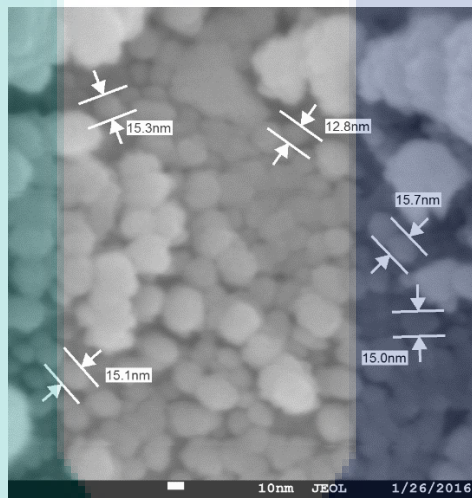


Figure 3.3 FESEM result of dry Al<sub>2</sub>O<sub>3</sub> nanoparticles at X300,000 magnification

Table 3.1 Properties of nanoparticles used in experiment

Property	Al <sub>2</sub> O <sub>3</sub>
Average nanoparticle size, nm	13
Molar mass, g·mol <sup>-1</sup>	101.96
Density, kg·m <sup>-3</sup>	4000
Thermal conductivity, W·m <sup>-1</sup> ·K <sup>-1</sup>	36
Specific heat capacity, J·kg <sup>-1</sup> ·K <sup>-1</sup>	880

Source: Lim et al. (2016)

Mixture of water-ethylene glycol as base fluid was used to prevent the galvanic corrosion of the cooling channel system and to prevent distilled water from freezing or boiling at extreme temperature. Peyghambarzadeh et al. (2011) stated that the mixture percentage of water and ethylene glycol in 40%:60% does not freeze at -45 °C because ethylene glycol has lower volatility compared to water. Table 3.2 shows the properties

of pure ethylene glycol in the liquid state (Peyghambarzadeh et al., 2011; Yu et al., 2012).

Table 3.2 Properties of Ethylene Glycol solution

Property	EG
Vapor pressure, mmHg at 20 °C	0.08
Boiling point, °C	195–198
Melting point, °C	-13
Density, g·ml <sup>-1</sup> at 25 °C	1.113

Source: Lim et al. (2016)

Pre-calculated mass of Al<sub>2</sub>O<sub>3</sub> nanoparticles by Equation 3.1 was dispersed in the base fluids with a two-step method proposed by Yu et al. (2012) and Eastman et al. (1997). The solution is diluted by Equation 3.2 from high to low concentration to prepare the nanocoolant of five volume concentrations with 0.2%, 0.4%, 0.6%, 0.8%, and 1.0%.

$$\phi = \frac{m_p / \rho_p}{V_p + V_{bf}} \times 100 \quad (3.1)$$

$$\Delta V = (V_2 - V_1) = V_1 \left( \frac{\phi_1}{\phi_2} - 1 \right) \quad (3.2)$$

where  $\phi$  is the volume concentration of nanocoolant;  $V_{bf}$  is the volume of base fluids; and  $m_p$ ,  $\rho_p$  and  $V_p$  are the mass of nanoparticles in SI unit of kg, the density of nanoparticles and volume of nanoparticles, respectively. The mixture of Al<sub>2</sub>O<sub>3</sub> nanocoolant is prepared using a magnetic stirrer. The dispersion stability of the nanocoolant improved after immersion in ultrasonic homogenizer model Fisherbrand FB15051 for one hour as shown in Figure 3.4. The stability of the nanocoolant is checked and confirmed after a month of preparation, as shown in Figure 3.5. A complete set of the nanocoolants sedimentation test with different volume concentrations of nanoparticles and three different ethylene glycol ratios could be viewed from Appendix C.



Figure 3.4  $\text{Al}_2\text{O}_3$  nanocoolant immersed in ultrasonic bath heater

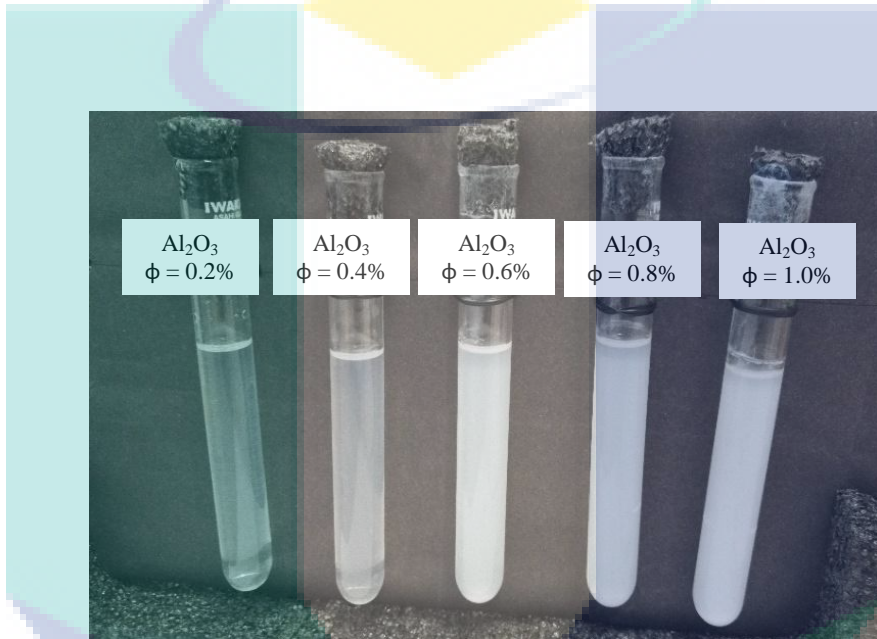


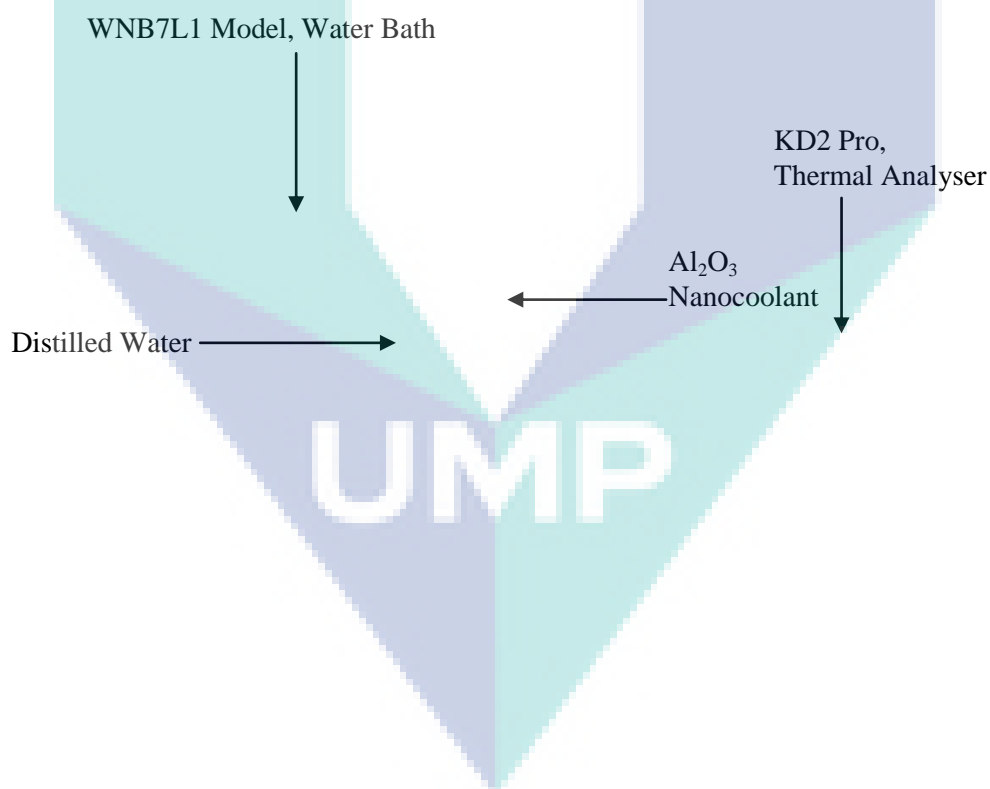
Figure 3.5 Nanocoolant samples of  $\text{Al}_2\text{O}_3$ /ethylene glycol-water mixtures after a month of preparation

### 3.3 Thermal Conductivity Measurement

KD2 Pro thermal property analyser made from Decagon Devices, USA was used to measure the thermal conductivity of the nanocoolant, as illustrated in Figure 3.6. The thermal physical properties of liquids and solids were determined by the device using the transient line heat source. The device meets the standards of both IEEE 442-1981 and ASTM D5334.

A single needle sensor named as KS-1 in the range of  $0.002$  to  $2.00 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  was used, and the sensor was validated by measuring the thermal conductivity of the verification liquid such as Glycerine, that was provided by the supplier. The measured

value of Glycerine at 25 °C was  $0.286 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , which agreed with the calibrated data of  $0.285 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  and within  $\pm 0.4\%$  accuracy (Zakaria et al., 2015). The validation process of the sensor was checked before each measurement of thermal conductivity. A water bath of WNB7L1 model by Memmert was used to maintain a constant temperature of the sample with an accuracy of 0.1 °C (Azmi et al., 2013). The thermal conductivity of 0.2 to 1.0% volume concentrations of aluminium oxide ( $\text{Al}_2\text{O}_3$ ) and the nanocoolant was measured within the temperature range of 15 to 55 °C. The consistency of data measurement was ensured by taking a minimum of three values for every concentration at a specific temperature, and the average of the three values was analysed. The thermal conductivity models (Hamilton and Crosser, 1962; Timofeeva et al., 2007; Wasp et al., 1977; Yu and Choi 2003) are shown in Table 3.3 and used to verify the experimental results of the thermal conductivity of nanocoolant.



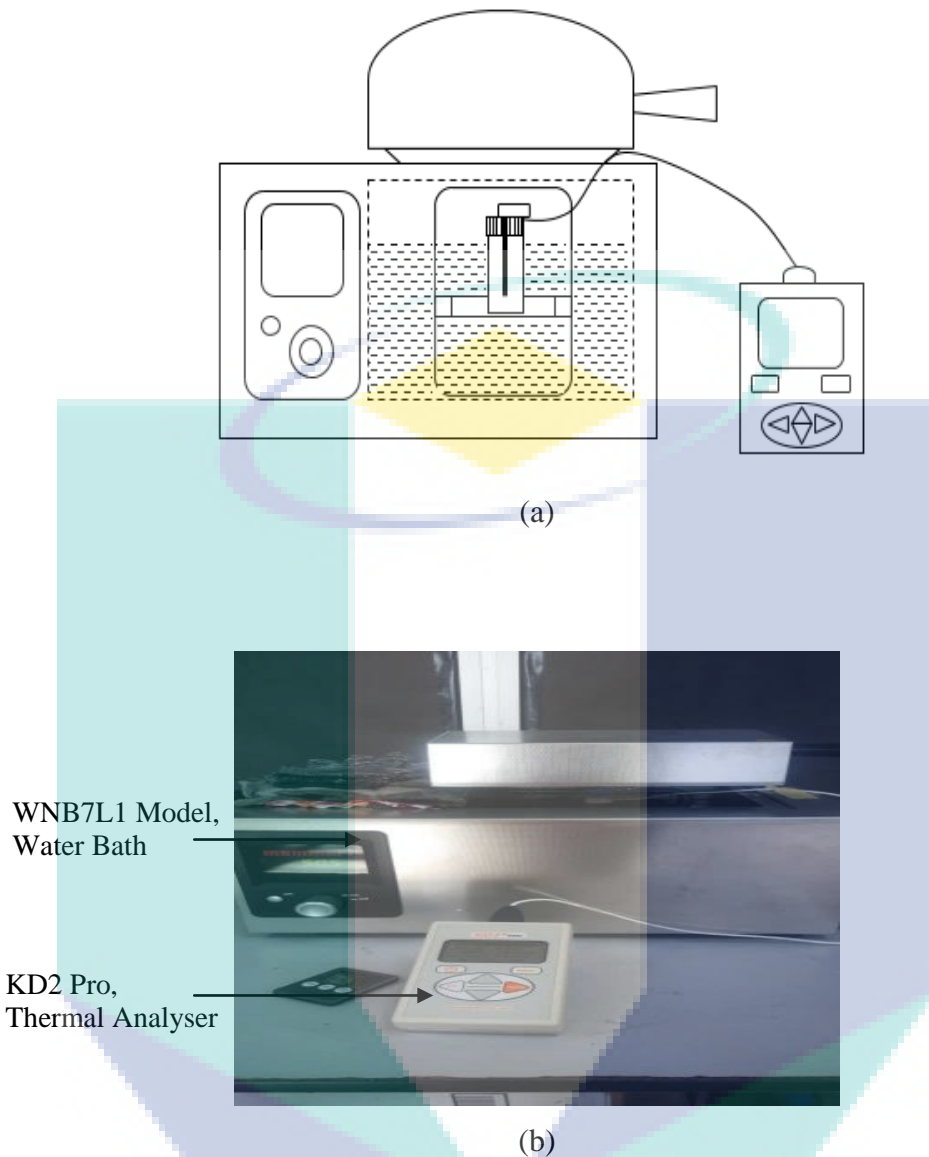


Figure 3.6 (a) Schematic diagram of thermal conductivity measurement; (b) Experiment setup for thermal conductivity measurement.

Table 3.3 Thermal conductivity models for nanocoolant

Model	Thermal Conductivity
Hamilton and Crosser (1962)	$k_r = \frac{k_{eff}}{k_{bf}} = \left[ \frac{k_p + (n-1)k_{bf} - (n-1)(k_{bf} - k_p)\varphi}{k_p + (n-1)k_{bf} + (k_{bf} - k_p)\varphi} \right],$ <p style="text-align: center;"><math>n = 3 \Rightarrow</math> spherical shape</p>
Timofeeva et al. (2007)	$k_r = \frac{k_{eff}}{k_{bf}} = (1 + 3\varphi)$



Wasp et al. (1977)	$k_r = \frac{k_{eff}}{k_{bf}} = \left[ \frac{k_p + 2k_{bf} - 2(k_{bf} - k_p)\varphi}{k_p + 2k_{bf} + (k_{bf} - k_p)\varphi} \right]$
Yu and Choi (2003)	$k_r = \frac{k_{eff}}{k_{bf}} = \left[ \frac{k_p + 2k_{bf} + 2(k_p - k_{bf})(1 + \beta)^3 \times \varphi}{k_p + 2k_{bf} - (k_p - k_{bf})(1 + \beta)^3 \times \varphi} \right], \beta = 0.1$

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### 3.4 Dynamic Viscosity Measurement

Dynamic viscosity of 0.2 to 1.0% volume concentrations of aluminium oxide ( $Al_2O_3$ ) in three different ratios of base fluids was measured by Brookfield which is a low viscosity digital viscometer with LVDV-III Ultra Programmable Rheometer, as depicted in Figure 3.7. The device is equipped with a personal computer for data collection and storage. The viscometer measures the dynamic viscosity of aqueous solutions that range from 1 to 6,000,000 mPa's by utilizing an ultra-low adapter. The spindle connected to the viscometer was used to measure nanocoolants dynamic viscosity (Azmi et al., 2014).

The viscometer drives a spindle that was immersed in nanocoolants. Spindle rotation created a viscous drag of the fluid opposite to the spindle, which was measured by the deflection of the calibrated spring. The adapter in this experiment had a provision for temperature circulation of bath fluid. The dynamic viscosity of different volume concentrations of the  $Al_2O_3$  nanocoolant started from 15 to 55 °C at an interval of 5 °C. Each measurement was conducted three times to generate a reliable data, and the average value of the three values was considered for analysis. The dynamic viscosity models (Pak and Cho, 1998; Wang et al., 1999; Sundar et al., 2012; Batchelor, 1977) are listed in Table 3.4 and used to compare the measured values of dynamic viscosity at different volume concentrations.

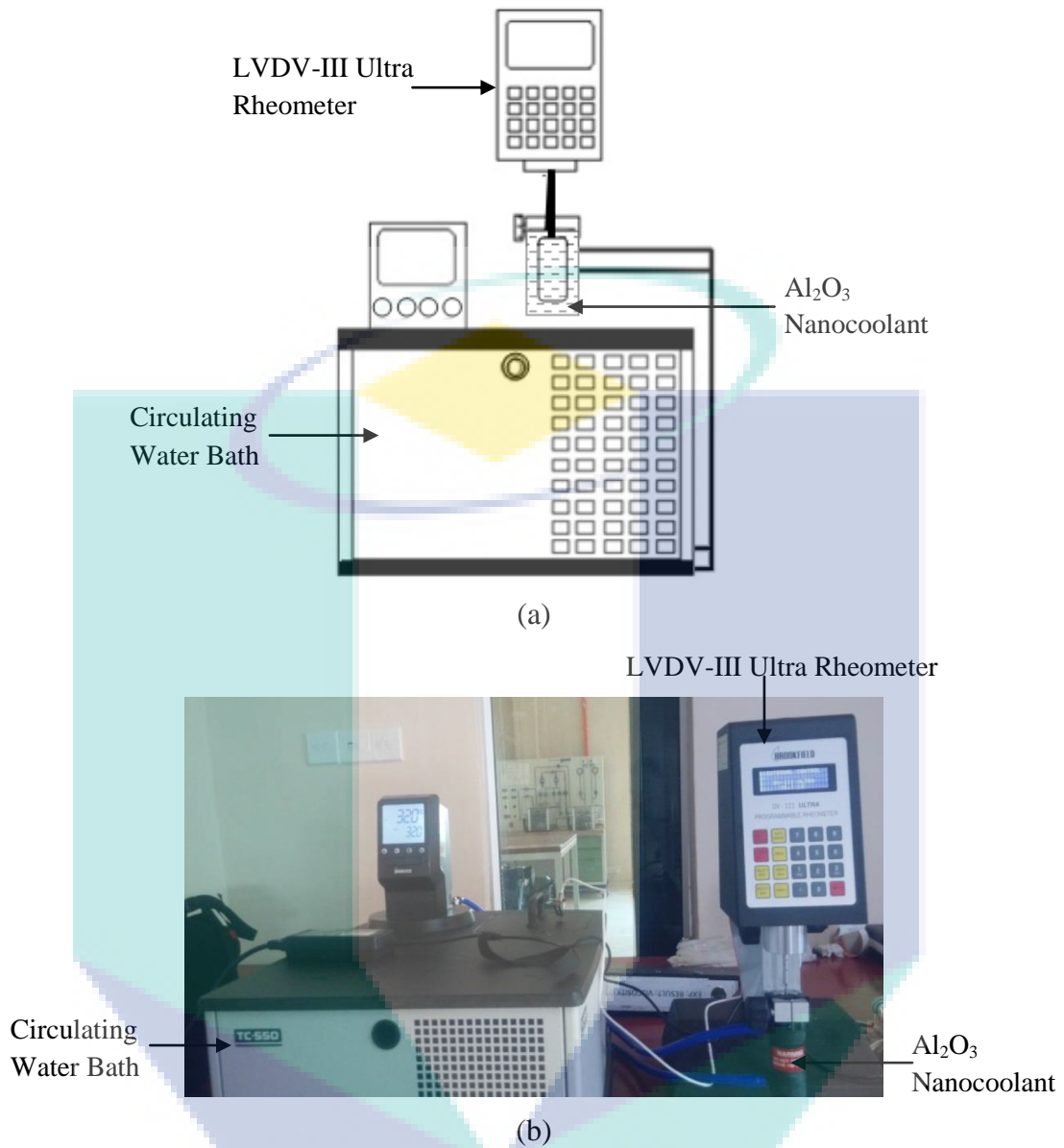


Figure 3.7 (a) Schematic diagram of dynamic viscosity measurement; (b) Experiment setup for dynamic viscosity measurement

Table 3.4 Dynamic viscosity models for nanocoolant

Model	Dynamic Viscosity
Pak and Cho (1998)	$\mu_r = \frac{\mu_{eff}}{\mu_{bf}} = 533.9\varphi^2 + 39.11\varphi + 1$
Wang et al. (1999)	$\mu_r = \frac{\mu_{eff}}{\mu_{bf}} = 1 + 7.3\varphi + 123\varphi^2$
Sundar et al. (2012)	$\mu_r = \frac{\mu_{eff}}{\mu_{bf}} = Ae^{B\varphi}, A = 0.9299 \text{ \& } B = 67.43 \Rightarrow 60 : 40\% (W / EG)$
Batchelor (1977)	$\mu_r = \frac{\mu_{eff}}{\mu_{bf}} = 1 + 2.5\varphi + 6.5\varphi^2$

### 3.5 Results and Discussion

In this section, the results of thermal conductivity, dynamic viscosity and convective heat transfer of aluminium oxide ( $\text{Al}_2\text{O}_3$ ) were obtained and discussed with several mathematical models. The nanocoolant after preparation by using two-step method and uniformly dispersed, whilst for thermal physical properties evaluation. The findings of the experimental data would be adapted into convective heat transfer analysis via mathematical equations and models. The relationship between thermal conductivity to bulk temperature; dynamic viscosity to bulk temperature; and variation of heat transfer distribution as function of Reynolds number was investigated.

#### 3.5.1 Thermal conductivity of nanocoolant

Figure 3.8 shows the thermal conductivity of the aluminium oxide ( $\text{Al}_2\text{O}_3$ ) nanocoolant in the ratio 60%:40% of water to ethylene glycol (W/EG) base fluid at 25 °C for 0.2 to 1.0% volume concentrations. The experimental results of the present study were compared with the estimated values obtained from previously published models. The figure shows that the thermal conductivity of the nanocoolant increased with volume concentration. The experimental values for this study were slightly higher than those of the three models by Hamilton and Crosser (1962); Timofeeva et al. (2007); and Wasp et al. (1977). However, the model by Yu and Choi (2003) agreed with the experimental values to a certain extent. The mean and maximum deviation of the three different ratio of water to ethylene glycol base fluids such as 60%:40%, 50%:50%, and 40%:60% experimental values are compared with the findings of Yu and Choi (2003) are 0.01% and 0.13%; 0.04% and 0.68%; and 0.03% and 1.44%, respectively. Elias et al. (2014) compared the experimental value of thermal conductivity of the  $\text{Al}_2\text{O}_3$ /radiator nanocoolant with the Hamilton and Crosser (1962) model. The results showed that their experimental value exhibited a 2.2% deviation from the calculated model. Murshed et al. (2008) used the Wasp et al. (1977) model that yielded a similar outcome as the Maxwell model for spherical nanoparticles and found that the experimental thermal conductivity values of  $\text{TiO}_2$ /water based nanocoolant showed 17.0% deviation from the Wasp model.

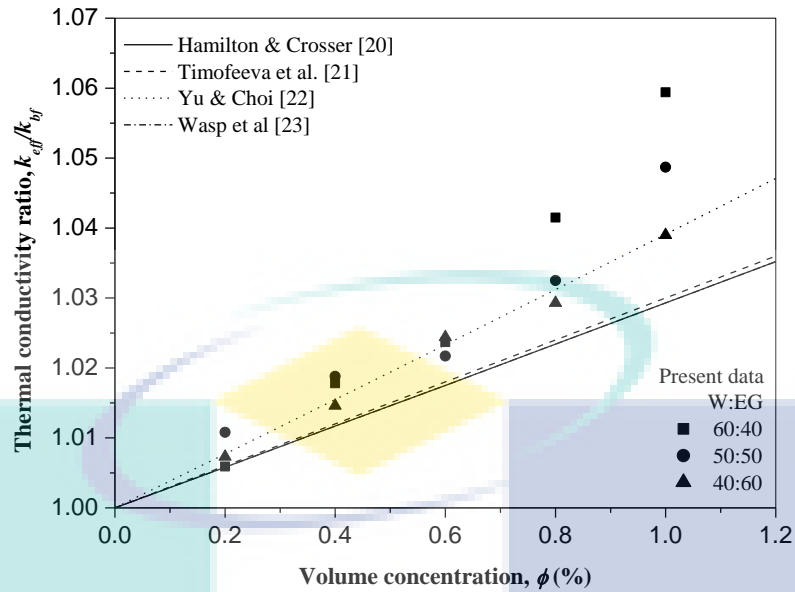


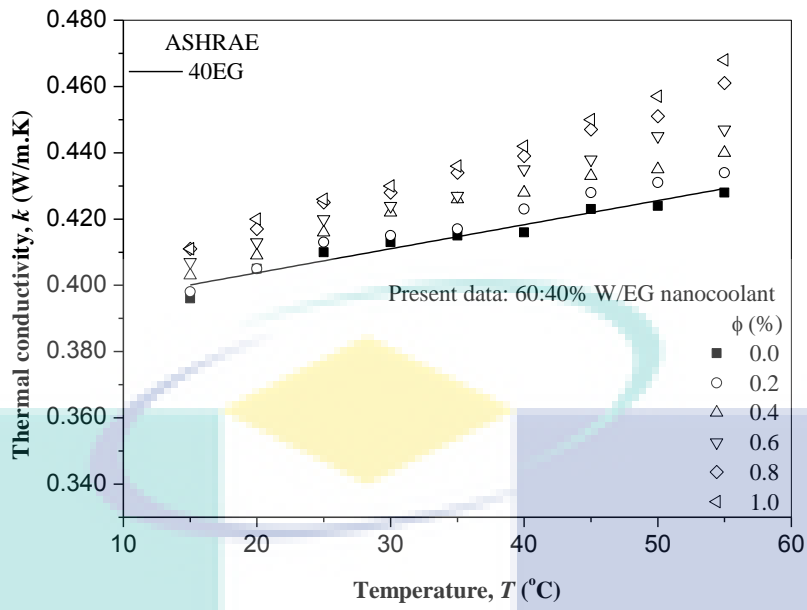
Figure 3.8 Variation of thermal conductivity ratio as function of nanoparticles volume concentrations in W/EG mixture at 25 °C

Figure 3.9 shows the thermal conductivity values of three different ratio of base fluids namely 60%:40%, 50%:50%, and 40%:60% water-ethylene glycol (W/EG) and compared with the values obtained from the ASHRAE handbook (2006). The experimental values of all fluids exhibited  $\pm 1.0\%$  deviation with handbook data in the measured temperature range. The nanocoolant with 0.2 to 1.0% volume concentrations were tested with the thermal conductivity instrument. Figure 3.9(a) represents the obtained data of 60%:40% (W/EG) nanocoolant with the data of the base fluid. The figure shows that the thermal conductivity of the nanocoolant increases when temperature and volume concentration are increased. The highest achievable thermal conductivity is  $0.468 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  at 1.0% volume concentration and 55 °C. The thermal conductivity enhancement ratio of the nanocoolant is 1.1 times higher when compared with the base fluid under the same temperature.

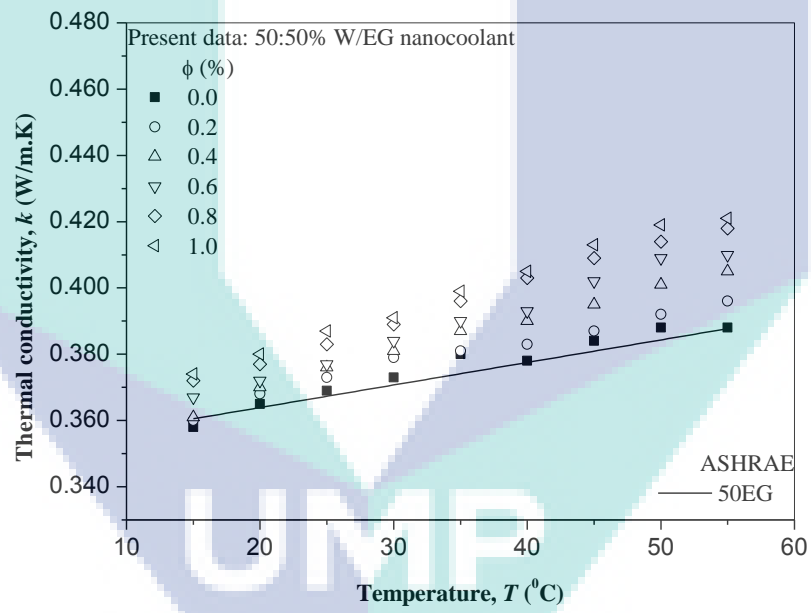
Figure 3.9(b) shows the experimental thermal conductivity values of the 50%:50% (W/EG) base fluid. The thermal conductivity increases with the increase of volume concentration and temperature. The thermal conductivity of the nanocoolant with 1.0% volume concentration at 55 °C is  $0.421 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ . Thermal conductivity enhancement ratio is 1.07 times at 55 °C and 1.0% volume concentration compared with the base fluid. The experimental thermal conductivity data of 40%:60% (W/EG) base fluid nanocoolant is shown in Figure 3.9(c). A similar thermal conductivity of the

nanocoolant trend with increased volume concentrations and temperature were obtained. Thermal conductivity enhancement ratio at 1.0% volume concentration of  $\text{Al}_2\text{O}_3$  is 1.08 times compared with the base fluid at 55 °C. Experimental thermal conductivity value under the same temperature and same volume concentration of nanoparticles is  $0.385 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ . The pattern agrees well with the trend of thermal conductivity enhancement from earlier reported studies (Elias et al., 2014; Sundar et al., 2014; Azmi et al., 2016).

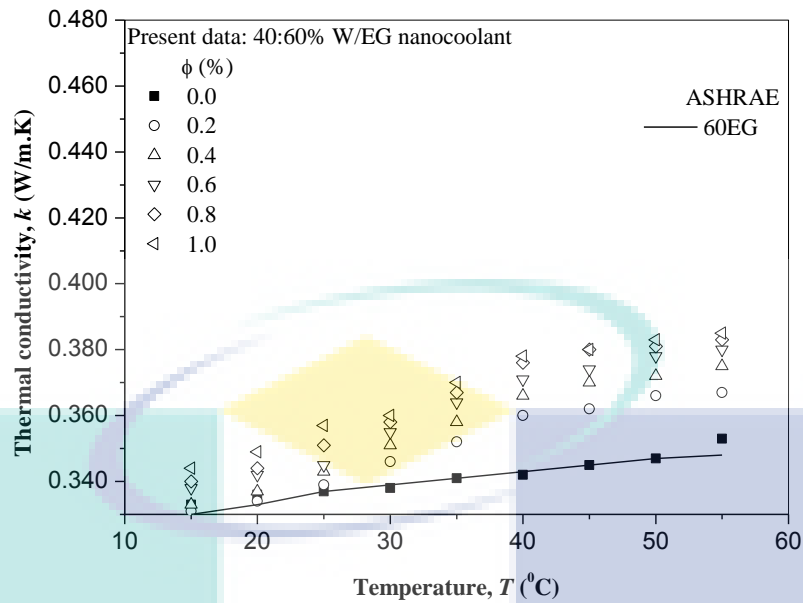
Thermal conductivity augmentation is due to the convection heat transfer of nanoparticles and Brownian motion in the base fluids. The thermal conductivity enhancement also depends on the effect of the base fluid (Azmi et al., 2016). The 60%:40% (W/EG) based nanocoolant has better enhancement of thermal conductivity compared with 50%:50% and 40%:60% (W/EG) based nanocoolants. Water is a better thermal conductivity fluid compared with ethylene glycol. A high percentage of EG addition into water reduces the thermal conductivity of water to obtain less thermal conductivity value. The thermal conductivity of 60%:40% (W/EG) base fluid attained the highest experimental value of  $0.410 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  at 25 °C. However, 50%:50% and 40%:60% (W/EG) base fluids are  $0.369 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  and  $0.337 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , respectively. Therefore, the thermal conductivity of the nanocoolant increases with the increase of temperature and volume concentration, but the increase depends on the base fluid. Moreover, the thermal conductivity enhancement of the  $\text{Al}_2\text{O}_3$  nanocoolant is significant with more than 5% for the volume concentration of higher than 0.6%.



a) 60%:40% (W/EG)



b) 50%:50% (W/EG)



c) 40%:60% (W/EG)

Figure 3.9 Thermal conductivity of different nanoparticle concentrations for three different weight of W/EG mixture base fluids

### 3.5.2 Dynamic viscosity of nanocoolant

Dynamic viscosity of aluminium oxide,  $\text{Al}_2\text{O}_3$ /water-ethylene glycol (W/EG) mixture as nanocoolants for 0.2 to 1.0% volume concentrations and 25 °C has been presented in Figure 3.10. The figure shows that the nanocoolant dynamic viscosity increased exponentially with the increment of volume concentration. The models of Pak and Cho (1998); Wang et al. (1999); Sundar et al. (2012); and Batchelor (1977) were utilized to compare with the present data for different volume concentrations at 25 °C. The models of Wang et al. (1999); and Batchelor (1977) largely under predicted the nanocoolants dynamic viscosity. However, the model by Pak and Cho (1998) agrees with the experimental values of 50%:50% and 40%:60% (W/EG) based nanocoolants. The mean and maximum deviation of experimental values compared with that of Pak and Cho are 0.37% and 5.23% for 50%:50% (W/EG); and 0.76% and 7.6% for 40%:60% (W/EG), respectively. The experimental values of 60%:40% (W/EG) based nanocoolant had a similar trend as the dynamic viscosity model of Sundar et al. (2012). The mean and maximum deviation of the experimental values compared with that of Sundar et al. (2012) are 0.74% and 14.35%, respectively.



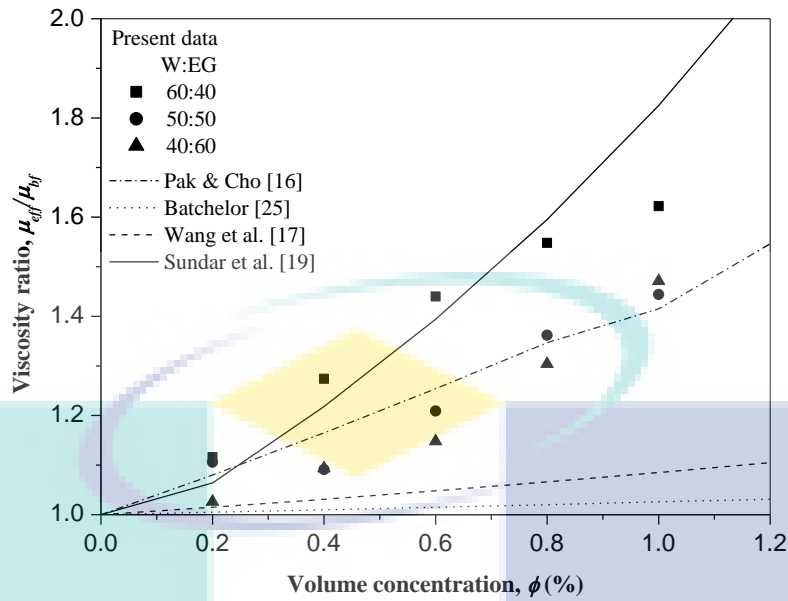


Figure 3.10 Variation of viscosity ratio as fraction of nanoparticle volume concentrations in W/EG mixture at 25 °C

Murshed et al. (2008) compared the experimental values of dynamic viscosity of  $\text{Al}_2\text{O}_3$ /water based nanofluids with that of Wang et al. (1999). The results showed that their experimental values were higher than those of Wang et al. (1999) by 86% deviation. However, Aladag et al. (2012) used the Batchelor model to calculate the dynamic viscosity of CNT nanofluids. They found that the dynamic viscosity of CNT nanocoolant experimental values showed 26% deviation from the Batchelor (1977) model. Kole et al. (2010) studied the dynamic viscosity of  $\text{Al}_2\text{O}_3$  in car engine coolant and compared their viscosity results with those of the Batchelor (1977); Brinkman (1952); and K-D model (Azmi et al., 2012). They found that the models failed to predict the measured dynamic viscosity of the nanocoolants. The measured dynamic viscosity values are unpredicted by the two models because of the difference in the size of the particle clusters and the differences in the dispersion techniques in the present study. The models of Wang et al. (1999) and Batchelor (1977) only considered volume concentration, but the nanoparticles can easily form clusters and experience surface adsorption in fluids. The relative dynamic viscosity is increased because the hydrodynamic diameter of nanoparticles increased by the clustering and adsorption of the nanoparticles.

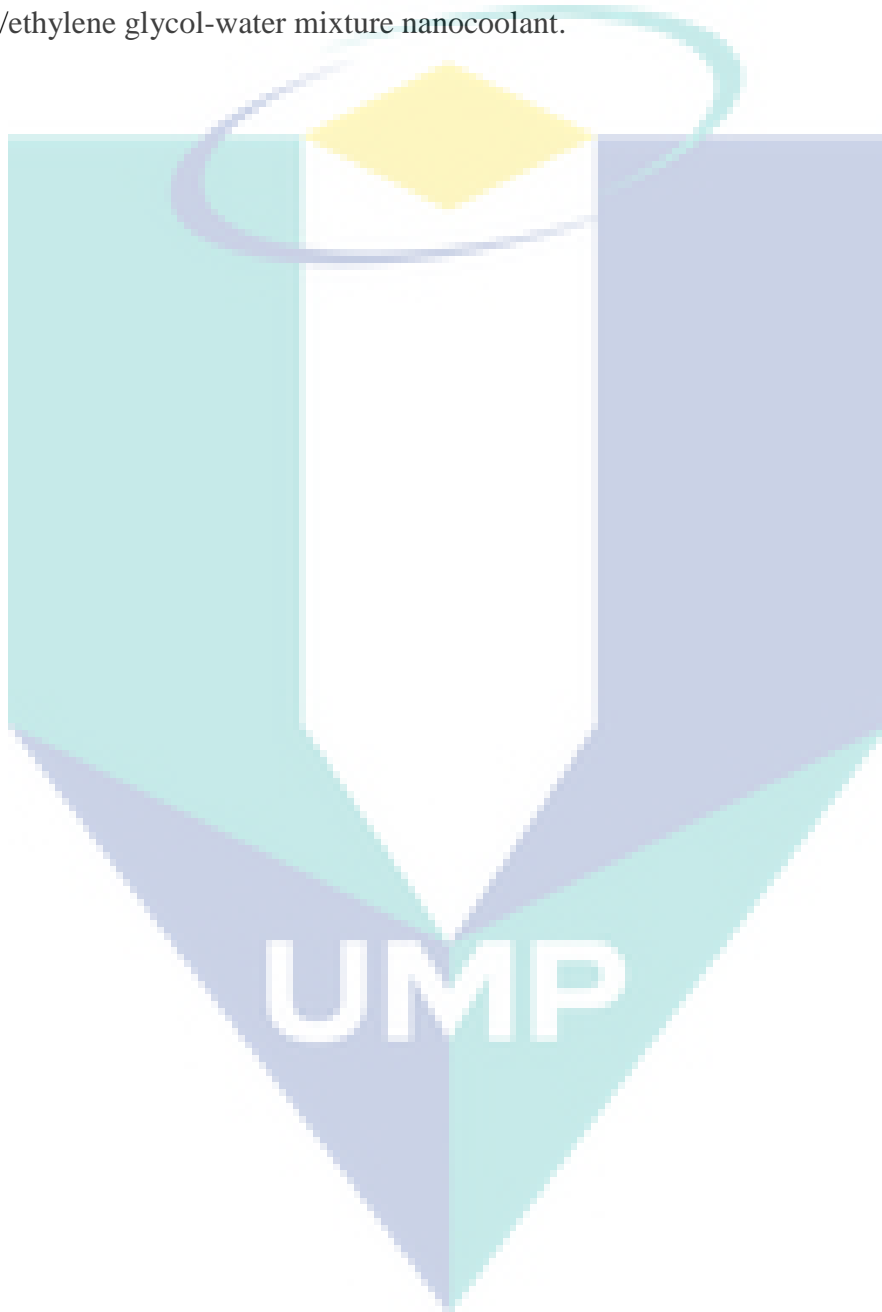
Figure 3.11 shows the dynamic viscosity values of three different ratio base fluids, such as 60%:40%, 50%:50%, and 40%:60% (W/EG) compared with the values

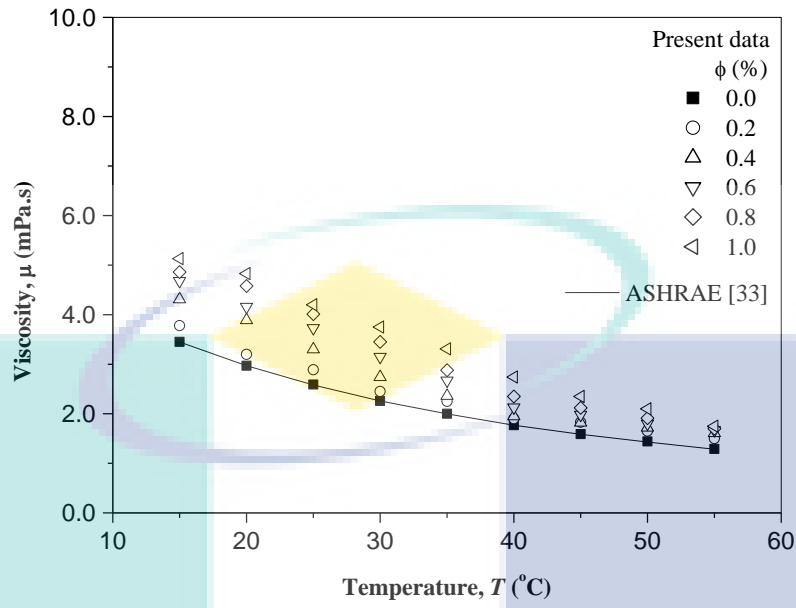
obtained from the ASHRAE handbook (2006). A maximum of  $\pm 9.0\%$  deviation was observed between the measured values of all fluids and the handbook data in the experimental range. The nanocoolants were verified in terms of Newtonian or Bringham-like behaviour. Moreover, the shear stress and shear strain rate of all the nanocoolants were observed. The shear strain rate of all the nanoparticle base fluids constantly increased with the increment of shear stress. Thus, nanocoolants exhibit Newtonian behaviour in the experimental temperatures and volume concentrations. A similar experiment of Newtonian behaviour has been investigated by Kole and Dey (2010) with  $\text{Al}_2\text{O}_3$ /engine oil nanocoolant, Aladag et al. (2012) by considering 50%:50% water mixture-based CNT nanocoolant and Namburu et al. (2007) with 40%:60% (W/EG) based CuO nanocoolant.

Figure 3.11(a) shows the experimental dynamic viscosity of  $\text{Al}_2\text{O}_3$  nanocoolant and 60%:40% (W/EG) base fluid. The figure shows that the dynamic viscosity of nanocoolants decreases with the increase of temperatures, but the increment of volume concentrations increases the dynamic viscosity compared with the base fluid. At 1.0% volume concentration, the dynamic viscosity enhancement ratio is 1.35 times at 55 °C compared with that of the base fluid. The experimental dynamic viscosity value of 50%:50% (W/EG) nanocoolant is shown in Figure 3.11(b) along with the base fluid. The dynamic viscosity enhancement ratio for 1.0% volume concentration is 1.23 times at 55 °C compared with that of the base fluid. Figure 3.11(c) shows the experimental dynamic viscosity values of 40%:60% (W/EG) base fluid. The dynamic viscosity enhancement of the nanocoolant with 1.0% volume concentration at 55 °C is 1.39 times compared with that of the base fluid.

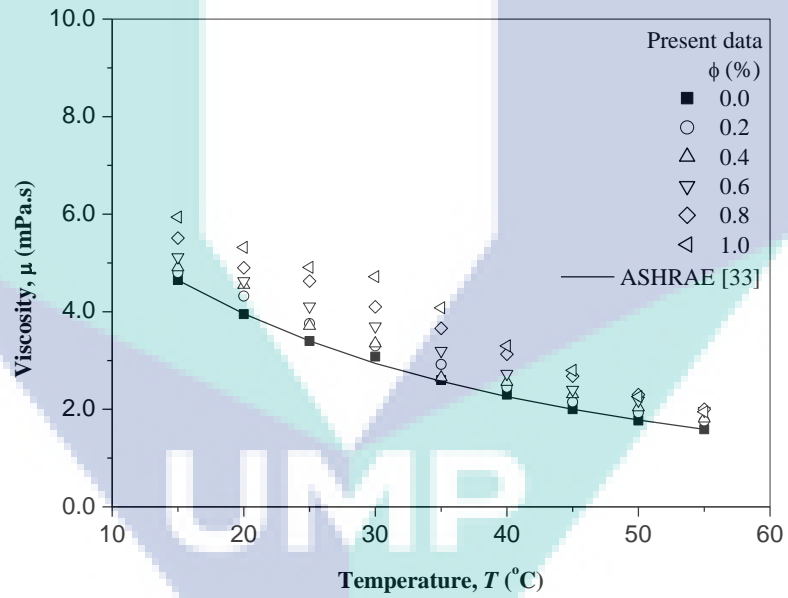
The enhancement of dynamic viscosity owing to the dispersion of nanoparticles in water-ethylene glycol mixture based  $\text{Al}_2\text{O}_3$  nanocoolant causes resistance between the fluid layers. Moreover, the present analysis for the dynamic viscosity of nanocoolant with the effect of base fluids shows that the 40%:60% (W/EG) based  $\text{Al}_2\text{O}_3$  nanocoolant provides more viscosity than the 50%:50% and 60%:40% (W/EG) based  $\text{Al}_2\text{O}_3$  nanocoolants in the measured volume concentrations and temperatures. The ASHRAE handbook (2006) also proves that the 40%:60% (W/EG) is more viscous than the 50%:50% and 60%:40% (W/EG) base fluids. Therefore, the high amount of ethylene glycol base fluid attains greater dynamic viscosity value compare with the low

amount of ethylene glycol base fluids. Sundar et al. (2014) detected the same behaviour of  $\text{Al}_2\text{O}_3$  nanoparticles dispersed in water and ethylene glycol. Their data showed that the ethylene glycol based nanocoolants were higher dynamic viscosity than water based nanocoolants. The results in Figure 3.11(a)-(c) shows that the enhancement of dynamic viscosity is insignificant for volume concentrations of less than 0.4% using the  $\text{Al}_2\text{O}_3$ /ethylene glycol-water mixture nanocoolant.

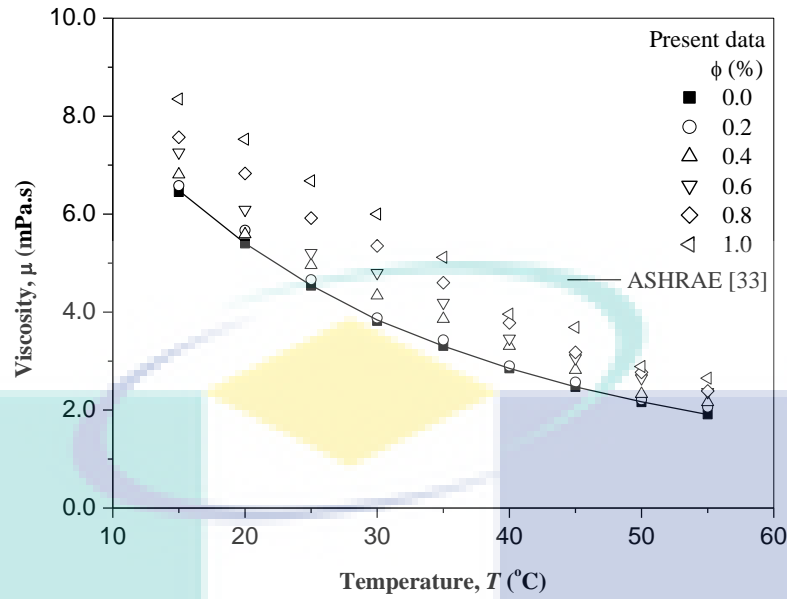




a) 60%:40% (W/EG)



b) 50%:50% (W/EG)



c) 40%:60% (W/EG)

Figure 3.11 Viscosity of different nanoparticle concentrations for three different weight of water-ethylene glycol mixture base fluids

### 3.5.3 Heat transfer coefficient of nanocoolant

The experimental data on thermal conductivity of the aluminium oxide,  $\text{Al}_2\text{O}_3$ /ethylene glycol-water mixture with volume concentration from 0.2 to 1.0% at three different base ratios is shown in Figure 3.12. The figure indicated that thermal conductivity increased with the decrease of the percentage of ethylene glycol mixed as the base fluid. Similar patterns agreed well with the findings of Sundar et al. (2013), who conducted the suspension of  $\text{Fe}_3\text{O}_4$  in ethylene glycol-water mixtures for three different base ratios and at higher temperature range. Azmi et al. (2013) analysed the condition that a nanocoolant does aid the improvement in heat transfer when the ratio of enhancement in dynamic viscosity to thermal conductivity compared to the base fluid which is lower than 4.0. The thermal physical properties evaluation in the present study concluded that with the use of  $\text{Al}_2\text{O}_3$  nanocoolant in 60%:40% (W/EG) based fluid will maximize the enhancement of thermal conductivity and simultaneously minimize the dynamic viscosity increment of nanocoolant. Therefore, the 60%:40% (W/EG) nanocoolant properties are advantages for heat transfer application and were used in the

further evaluation of heat transfer coefficient for hot press forming (HPF) cooling application.

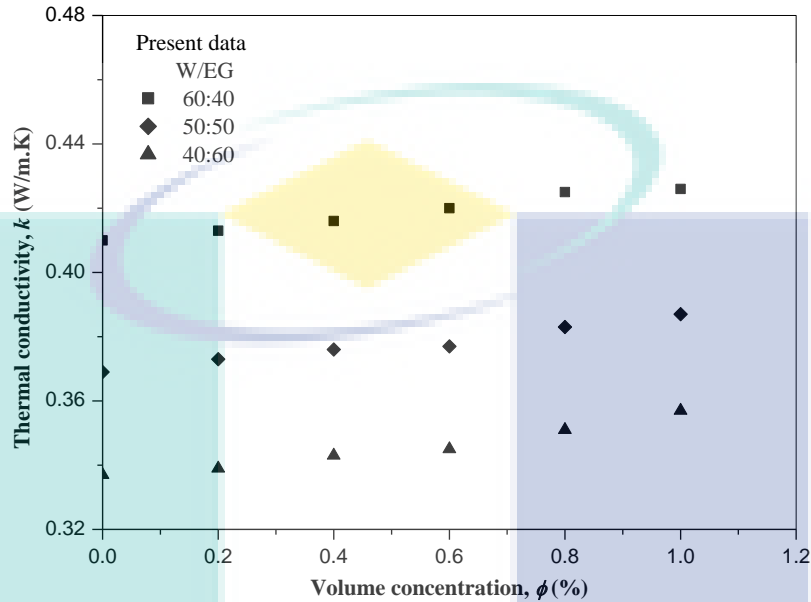


Figure 3.12 Relationship between thermal conductivity as function of nanoparticles volume concentration for three different base fluids

Nusselt number of base fluid 60%:40% of water-ethylene glycol mixture was estimated using equations of Lim et al. (2016). Chandrasekar et al. (2010); and Wen and Ding (2004) highlighted that larger Nusselt number caused the higher efficiency of convection process due to more suspended nanoparticles near the wall. Thus,  $\text{Al}_2\text{O}_3$  nanocoolant in 60%:40% water-ethylene glycol mixture base fluid could be implemented at volume concentration less than 1.0% and temperature of 25 °C for application in HPF die. The equation of Lim et al. (2016) is used for the estimation of the Nusselt number of  $\text{Al}_2\text{O}_3$  nanocoolants flow in a tube as shown in Equation 3.3. It is applicable for  $\text{Al}_2\text{O}_3$  nanocoolants with the different base ratio (BR) of water-ethylene glycol mixture at volume concentrations less than 1.0% and maximum temperatures of 70 °C. Further, the convective heat transfer coefficient is calculated from Equation 3.4.

$$Nu_{nf} = 0.025Re_{nf}^{0.76} Pr_{nf}^{0.45} \times \left[ (0.01 + BR)^{-0.1} \times \left( 1 + \frac{T_{nf}}{70} \right)^{0.14} \right] \quad (3.3)$$

$$h_{nf} = \frac{Nu_{nf} \times k_{nf}}{D} \quad (3.4)$$

Prandtl number of nanocoolant is obtained from Equation 3.5, while, the Reynolds number is obtained from Equation 2.1 in Section 2.9.2 with the properties referred at the bulk temperature,  $T_b$ .

$$Pr_{nf} = \frac{C_{nf} \mu_{nf}}{k_{nf}} \quad (3.5)$$

Thermal conductivity and dynamic viscosity for every concentration of  $Al_2O_3$  nanocoolants were investigated initially in the present work. Whereas, the density and specific heat of nanocoolant are determined by the classical model solid-liquid relation as in Equation 3.6 and Equation 3.7, respectively.

$$\rho_{nf} = \left(\frac{\phi}{100}\right)\rho_p + \left(1 - \frac{\phi}{100}\right)\rho_{bf} \quad (3.6)$$

$$C_{nf} = \frac{\left(1 - \frac{\phi}{100}\right)(\rho C)_{bf} + \left(\frac{\phi}{100}\right)(\rho C)_p}{\left(1 - \frac{\phi}{100}\right)\rho_{bf} + \left(\frac{\phi}{100}\right)\rho_p} \quad (3.7)$$

The heat transfer coefficient of  $Al_2O_3$  nanocoolant in 60%:40% (W/EG) at the temperature of 25 °C is higher compared to the base fluid as shown in Figure 3.13. At lower Reynolds number of 6,000, the heat transfer coefficient of the nanocoolant with 0.2% volume concentration is 4498.4  $W \cdot m^{-2} \cdot K^{-1}$  and it is enhanced by 2.9%. Besides, the heat transfer coefficient of 0.8% volume concentration is improved by 18.7% at the same Reynolds number compared to the base fluid. At higher Reynolds number of 20,000, the heat transfer enhancement of the nanocoolant at 0.2% and 0.8% volume concentrations are 4.0% and 24.9%, respectively. The convective heat transfer coefficient of 1.0% concentration in 60%:40% (W:EG) base fluid at 25 °C is enhanced by 25.4% better than that of 50%:50% and 40%:60% (W:EG) base fluid for Reynolds



number of 20,000. Figure 3.13 describes that with the addition of nanoparticles to water-ethylene glycol mixture base fluid will significantly improve the heat transfer coefficient of the nanocoolant. The similar pattern was also found by Yu et al. (2012). They stated that the increment of heat transfer performance is due to the increase of the effective thermal conductivity of nanocoolant. Therefore, the use of nanocoolant for application in the cooling channel of HPF die is significant to increase its heat transfer performance.

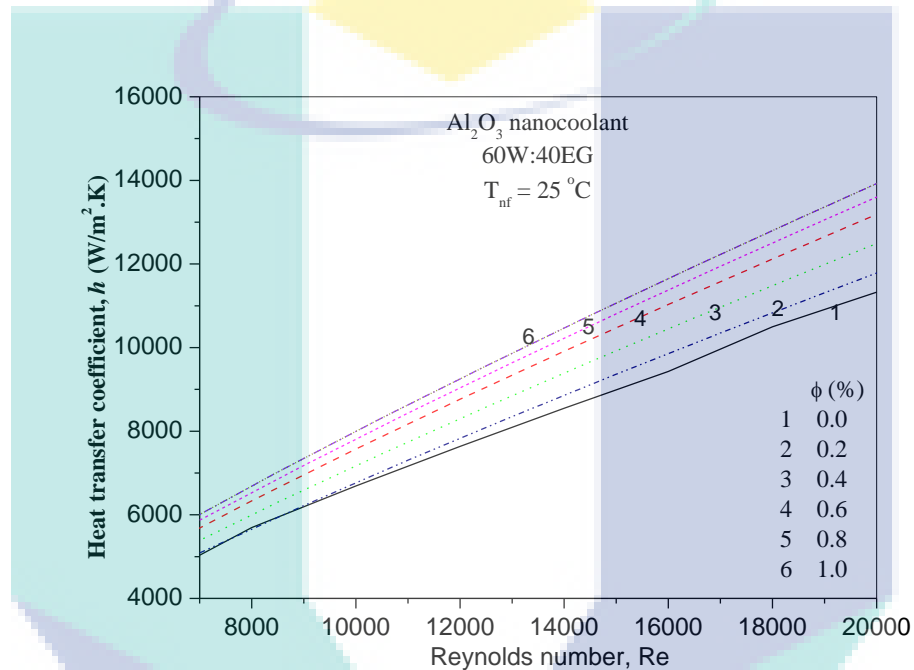


Figure 3.13 Distribution of heat transfer coefficient of 60%:40% water-ethylene glycol mixture based  $\text{Al}_2\text{O}_3$  nanocoolant

### 3.6 Summary

The thermal conductivity and dynamic viscosity of aluminium oxide,  $\text{Al}_2\text{O}_3$ /water-ethylene glycol mixture nanocoolants were investigated. The results show that thermal conductivity increases with the increment of volume concentrations and temperature. However, the thermal conductivity of nanocoolant decreases with the increase of ethylene glycol percentage in the base fluid. Temperature and volume concentration contribute significant effects on the effective thermal conductivity of nanocoolants. The experimental analysis found that dynamic viscosity of nanocoolants

increases with the increment of volume concentration. However, the dynamic viscosity of nanocoolants decreases with the increase of temperature.

The dynamic viscosity enhancement was investigated to be greater than the enhancement of thermal conductivity. The highest dynamic viscosity enhancement increased by 39% compared to the base fluid. However, the highest thermal conductivity enhancement increased by 10% compared with the base fluid. At 1.0% volume concentration, the heat transfer enhancement is 25.4% estimated for the nanocoolant bulk temperature of 25 °C. Therefore, finding the ideal volume concentration of Al<sub>2</sub>O<sub>3</sub>/water-ethylene glycol mixture nanocoolant is very important to increase the productivity of hot press forming (HPF) processes. The use of Al<sub>2</sub>O<sub>3</sub>/water-ethylene glycol mixture nanocoolants of 60%:40% (W/EG) with the volume concentration of less than 1.0% is recommended for application in the cooling system of HPF process. Further investigations and experiments on the performance of the HPF cooling system using Al<sub>2</sub>O<sub>3</sub>/ethylene glycol-water mixture nanocoolants are required to extend the present work in the next chapter.

The logo of UMPA (Universitas Mitra Bina Nusantara) is a large, stylized letter 'V' shape. The left side of the 'V' is light blue, the right side is light purple, and the bottom point is a darker blue. The letters 'UMPA' are written in white, bold, sans-serif font across the center of the 'V' shape.

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## CHAPTER 4

### EXPERIMENTAL INVESTIGATIONS OF NANOCOOLANT APPLICATION IN HOT PRESS FORMING PROCESS

#### 4.1 Introduction

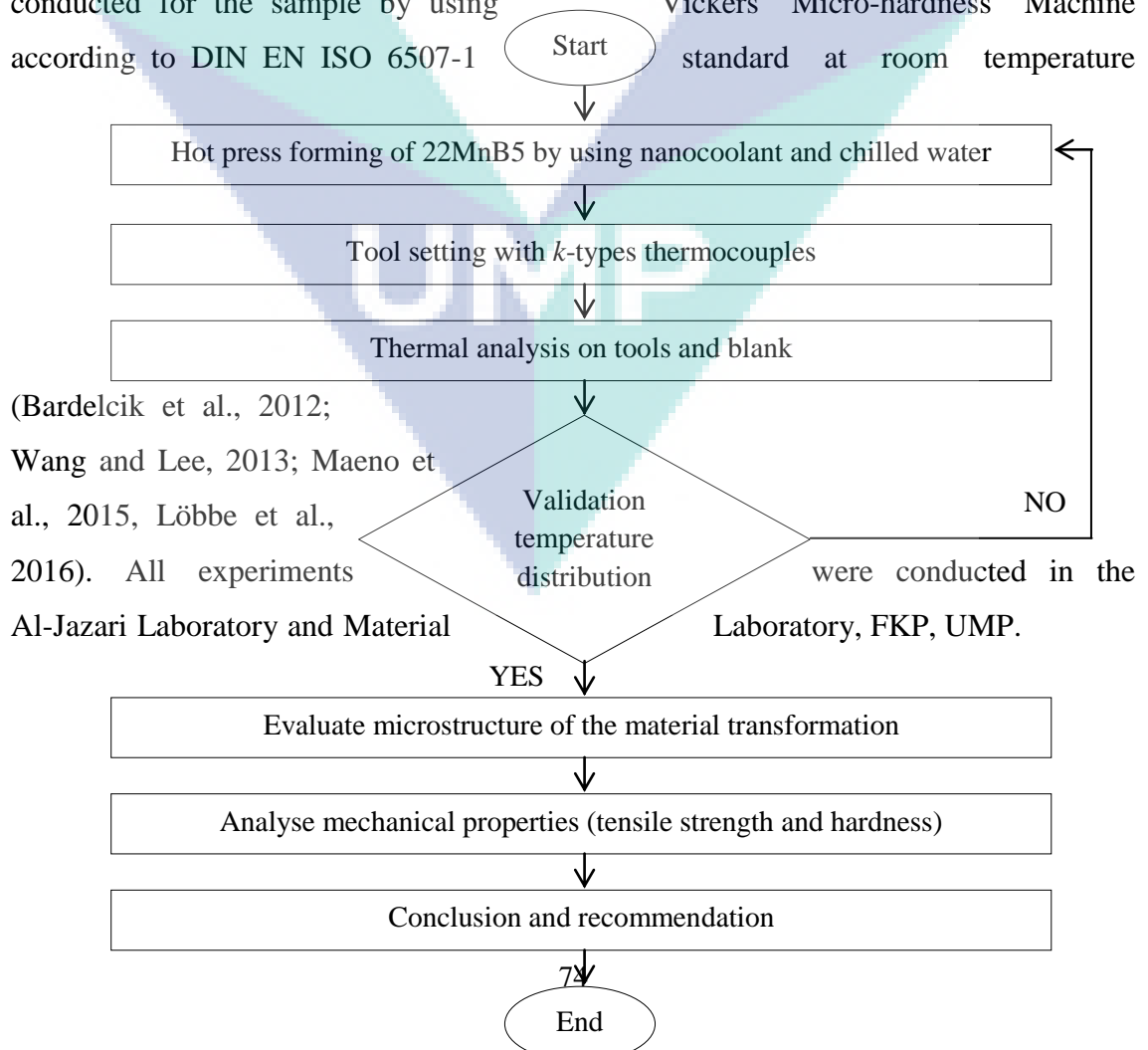
This chapter explained several methods which are used to achieve the aim and objectives of this research project. The sequence of the methods had been planned and applied for hat-shaped bending as shown in

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Figure 4.1. In previous Chapter 3, nanocoolants had been characterized in terms of thermal conductivity and dynamic viscosity and selected as the cooling agent to

introduce in the hot press forming (HPF) process experiment in this chapter. The experiment of HPF process started from the preparation of the hat-shaped boron steel blank by using cold forming die as pre-forming operation. The material used for the hat-shaped blank was boron steel with the grade of 22MnB5. Next, the nanocoolant was synthesized to a certain amount as cooling fluid for introducing it into the experiment. The methods of producing and homogenizing nanocoolant could be referred in previous Section 3.2. The *k*-type thermocouples were installed to the HPF dies to collect the temperature data during the HPF process (Kim et al., 2015). Meantime, two types of experiments such as nanocoolant and chilled water with constant flow rate and heat transfer experiment were conducted in this experimental study for investigating the heat transfer distribution of the HPF process.

Finally, the microstructure transformation of heated blanks were evaluated by using Light Optical Microscopy (LOM) (Naderi et al., 2011; Lbbe et al., 2016). The tensile tests were conducted according to American Society for Testing and Material International (ASTM E 8M) standard by using the Universal Tensile Machine (UTM) to find the ultimate tensile strength of the hot pressed part (Merklein et al., 2014; Mori et al., 2015). Once the tensile test had been performed, the hardness measurement was conducted for the sample by using Vickers Micro-hardness Machine according to DIN EN ISO 6507-1 standard at room temperature



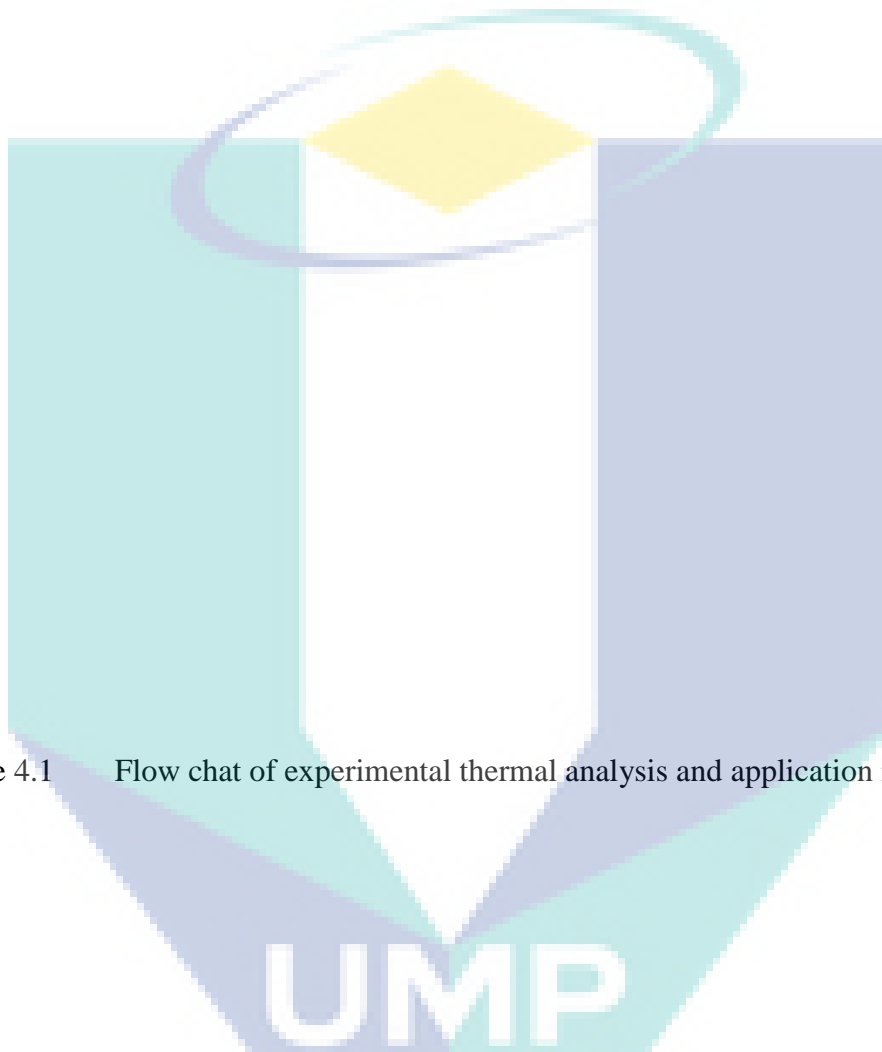


Figure 4.1 Flow chat of experimental thermal analysis and application in HPF

#### 4.2 Sample Preparation

Hat-shaped sample with tensile shape specification was used because the instrumentations in laboratory limited to hot press form the real automotive parts. The hat-shaped part was formed by using cold forming tool. The material used for the sheet metal blank product was boron steel with the grade of 22MnB5. While, SKD 61 was used as the hot press forming (HPF) tool material which can cover heat transfer distribution of the austenite sheet metal blank. Hat-shaped sample was chosen as the specimen of the experiment from Thanadngan et al., 2013 with the blank size dimension of 280 mm × 100 mm × 1.8 mm. Figure 4.2 concludes the process flow for

hat-shaped samples fabrication by using cold dies as pre-forming operation with a mechanical press machine model OCP 80.

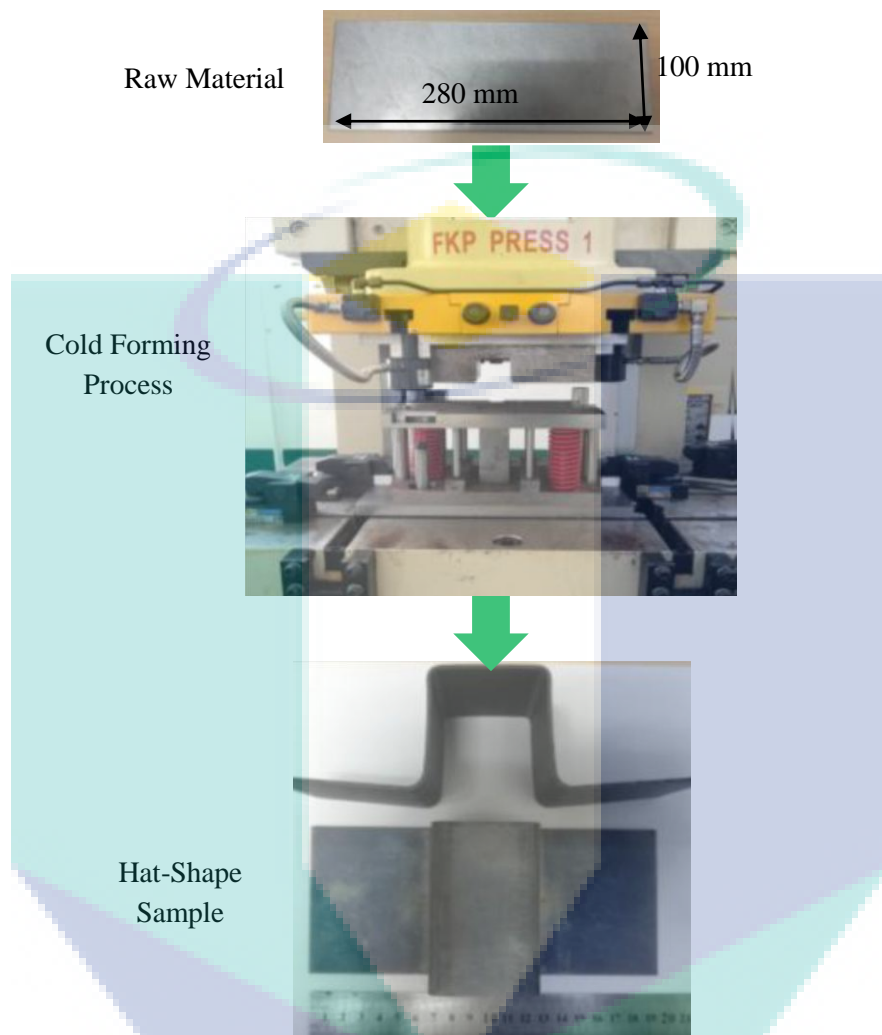


Figure 4.2 Pre-forming process of hat-shaped samples

#### 4.2.1 Nanocoolants as cooling fluid preparation

Regarding to previous Chapter 3 mentioned about the methods of nanocoolant preparation by using aluminium oxide ( $\text{Al}_2\text{O}_3$ ) nanoparticles suspended into the base fluid by using two-step technique with nanoparticles in powder form and later dispersed in the selected base fluid (Lim et al., 2016). The mass of  $\text{Al}_2\text{O}_3$  nanoparticles required was calculated by using Equations 3.1 in Section 3.2, subsequently by using Equation 4.6 which provided density properties. The nanocoolant for hot press forming (HPF) experiment was prepared in bulk quantities with volume of 20 litres. Figure 4.3 shows the process flow of preparation of  $\text{Al}_2\text{O}_3$  nanocoolants for investigation of heat transfer

distribution in HPF process. After the specific concentration of nanoparticles was calculated by Equation 3.1 and Equation 4.1, the  $\text{Al}_2\text{O}_3$  nanoparticles were weighted by using weighing machine in the material laboratory before being dispersed in the base fluid.

$$\phi = \frac{\frac{m_p}{\rho_p}}{\frac{m_p}{\rho_p} + \frac{m_{bf}}{\rho_{bf}}} \times 100 \quad (4.1)$$

Nanocoolant was prepared by synthesizing nanoparticles with base fluid by using mechanical stirrer. The dispersion of pre-calculated weight of  $\text{Al}_2\text{O}_3$  nanoparticles in water-ethylene glycol mixture was extended by immersing it in an ultrasonic homogenizer for one hour. Concerning to Suganthi and Rajan (2014), the sonication time period influenced the thermal physical properties of nanocoolants. They discovered that the nanocoolants obtained maximum thermal conductivity and also recommended to conduct sonication operation. Unfortunately, the prepared nanocoolant was homogenized regularly for 2 litres per cycle due to volume limitation of using an ultrasonic homogenizer. The sonication operation continued until 20 litres was completely homogenized and well mixed with a mechanical stirrer. As review from a group of researchers, Das et al. (2008) stated that nanocoolant was uniformly dispersed and well mixed was compulsory for effective production or reproduction of enhanced properties and interpretation of experimental data.



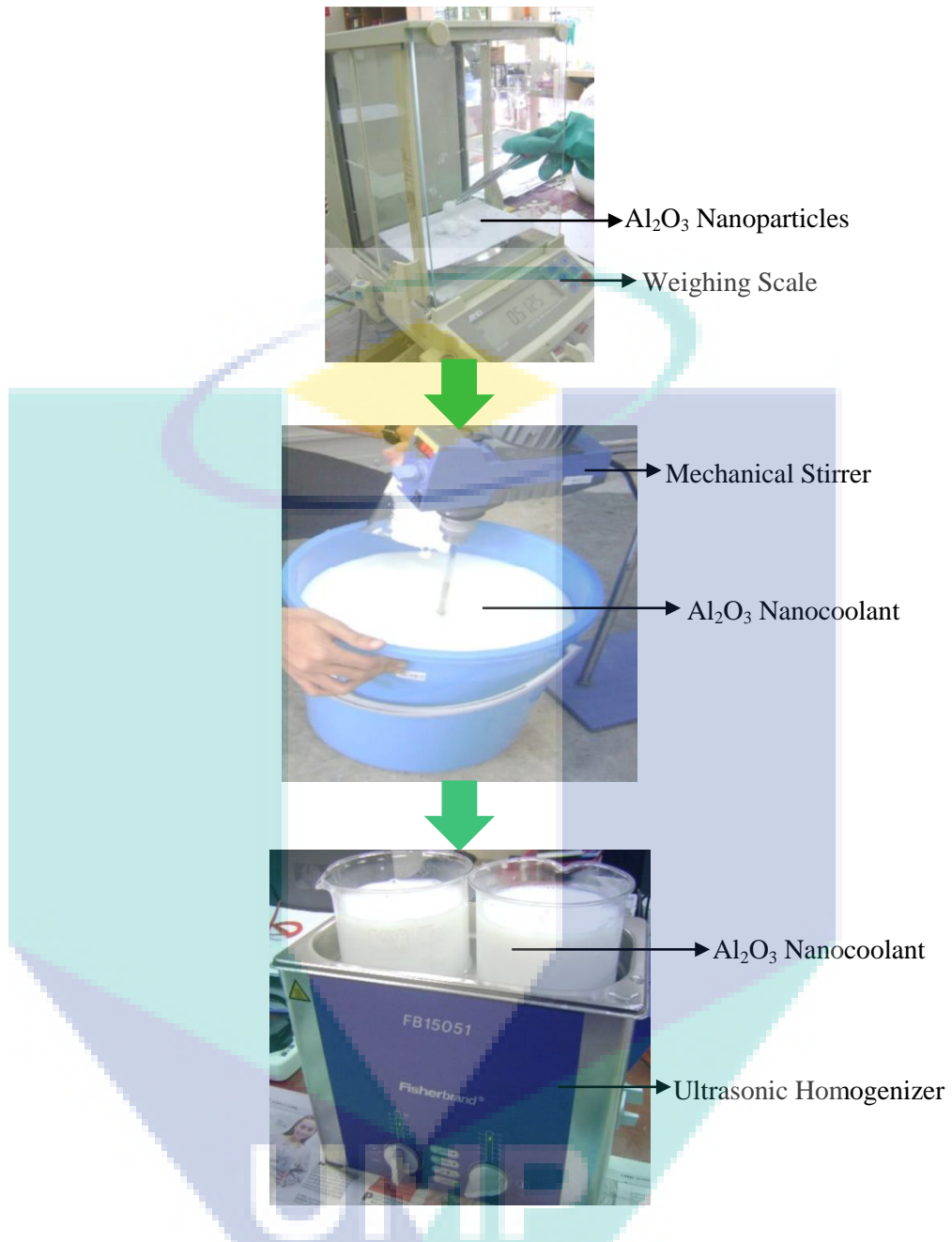


Figure 4.3 The preparation of Al<sub>2</sub>O<sub>3</sub> nanocoolant

In this research experiment, the nanocoolant was synthesized in volume concentration of 0.8% Al<sub>2</sub>O<sub>3</sub> nanoparticles were dispersed in the water-ethylene glycol based mixture with 60%:40% volume ratio as discussed in Chapter 3. The nanocoolant was synthesized in a volume of 20 litres for chiller operation. The reason of chiller was to maintain the bulk temperature of nanocoolant at around 25 °C for quenching operation in HPF process. There were no surfactants mixed in the preparation of

nanocoolants in accordance with Tony et al. (2013) which mentioned that by introducing surfactants, it might affect the thermal physical properties of nanocoolants. Serebryakova et al. (2015) and Zakaria et al. (2015) also synthesized their nanocoolants without adding surfactants and they found that nanocoolant were in a stable condition.

### 4.3 Thermocouples Setup

Thermocouples were located inside the hot press forming (HPF) dies, and *k*-type thermocouples were used in this experiment. Figure 4.4(a) depicts the *k*-type thermocouples placed at 6 different locations in HPF tool. The function of thermocouples was to measure the actual temperature changed during the quenching process in order to record the data to be analysed. George et al. (2012) and Lin et al. (2015) evaluated the heat transfer distribution of the tool by placing the thermocouple approximately in 4 mm away from the loading counter. The data of temperature changed was collected by using a data logger instrument of model ZR-RX25 Data Logger. The data logger device was used to measure a wide range of temperatures by using *k*-type thermocouples that have a miniature size thermocouple connector. Moreover, the data logger has low conversion time as 10 temperature measurements could be taken for every second. The location of thermocouple needed to be different from other researcher's work, since the data of the heat transfer distribution enabled to attain. In this experiment, the best location to collect the temperature of HPF dies was 0.05 mm away for the loading counter as shown in the Figure 4.4, and the 4 mm range enabled to get the desired distribution of temperature.

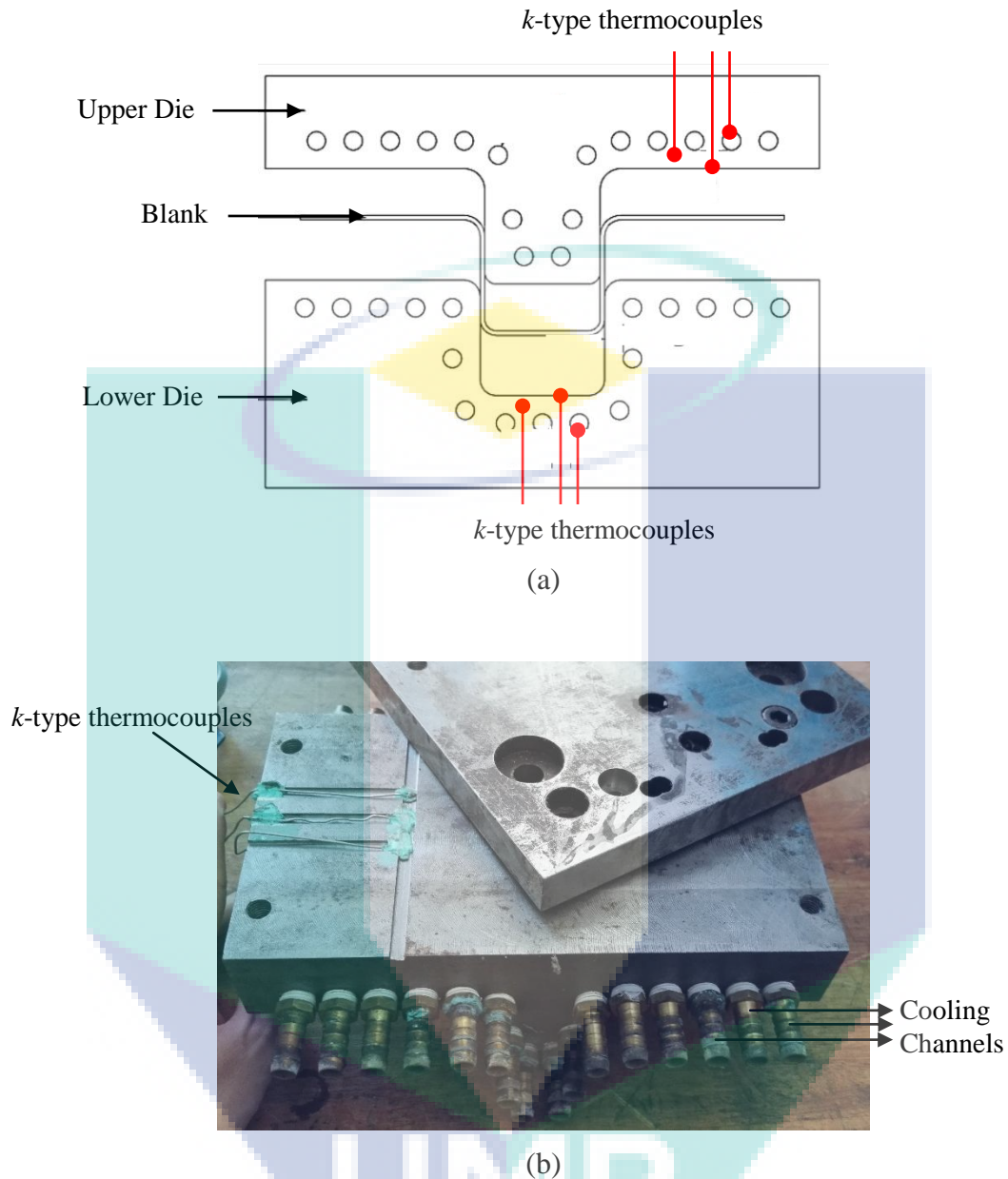


Figure 4.4 Thermocouples location in HPF tool a) Schematic diagram b) Three thermocouples in upper tool

#### 4.4 Hot Press Forming of Boron Steel Sheet Metal

Hot press forming (HPF) is a process to increase the tensile strength of boron steel by heating it up to austenite transformation temperature and instantly quenched it inside a coolant-cooled die to perform the part drawing (Hu et al., 2013). Several researchers Hoffmann et al. 2007; Karbasian et al. 2010 and Naganathan et al. 2012 had elaborated on the HPF process. The sheet metal was heated up to approximately 900 °C

for 5 mins heating time and then placed in an enclosure cooled die. The cooling channel has a convection heat transfer coefficient of  $7813 \text{ W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$  of nanocoolant with a bulk temperature of  $25 \text{ }^\circ\text{C}$ . Besides that, chilled water with  $4700 \text{ W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$  of convection heat transfer coefficient also used to cool down the dies and to allow the quenching operation to transpire.

After attaining the optimum heat transfer distribution data of HPF dies and blank from finite element analysis (FEA), the nanocoolant as a cooling agent of HPF process was synthesised prior to introduction into the experiment. The experiments of comparing two different cooling fluids as coolant with constant flow rate and temperature changed of heat distribution were conducted in this research study. The nanocoolant as an alternative cooling agent was used to compare with chilled water to determine the mechanical properties and microstructure of the hot pressed part. Merklein et al. (2008) and Wu et al. (2014) conducted an experiment to determine the mechanical properties in terms of tensile strength and hardness of the hot pressed part by using two different types of cooling fluid such as water and chilled water. Furthermore, the heat transfer distribution experiment was performed to evaluate the cooling rate of the HPF dies.

As for the preparation of sheet metal blank, mechanical press machine of model OCP 80 was used to fabricate the hat-shaped sample through the cold forming process as shown in Figure 4.5. After sheet metal blank preparation, hydraulic press machine as shown in Figure 4.6, which was used in conducting the HPF process experiment. Besides that, the specification of the mechanical press machine and hydraulic press machine were summarized in Table 4.1 and Table 4.2, respectively.

Table 4.1 The features of mechanical press machine model OCP 80

<b>Specifications</b>	<b>Values</b>
Max Die Height (mm)	330
Slide Area (mm X mm)	450 X 550
Stroke Length (mm)	150
Tonnage Capacity (Tonne)	80



Figure 4.5 Fabrication of hat-shape sample by using mechanical press machine, OCP 80

Table 4.2 The features of hydraulic press machine

Specifications	Values
Bore Diameter Size (mm)	80
Rod Diameter Size (mm)	40
Stroke Length (mm)	200
Tonnage Capacity (Tonne)	10



Figure 4.6 Hydraulic press machine used in hot forming operation

#### 4.4.1 Experimental study of temperature distribution

Current research project experiments studied several parameters that were used for hot press forming (HPF) process. These parameters such as austenitization temperature, cooling flow rate, pressure and quenching time were used to perform the part drawing in HPF process. As for the cooling flow rate, the value was fixed according to the power of motor water pump used in Acson model AMAC 40C chiller. Besides that, the thickness of sheet metal blanks was 1.8 mm and formed to hat-shaped sample through cold forming process. In this experiment, the hat-shaped blank was heated up to 900 °C of temperature for approximately 5 mins time period (Karbasian et al., 2010). Mori et al. 2009 and Lechler et al. 2008 found that 5 mins of heating time was sufficient to attain the optimum martensitic content in the quenched samples with a maximum hardness of approximately 470 HV. As for the quenching time during HPF process, the time interval was 3, 5, 8 and 10 s, respectively.

Figure 4.7 presents the experimental setup for the HPF process with chiller and heating furnace. The furnace was used to heat up the boron sheet metal blank to the austenitization temperature and the chiller was used to cool down the HPF die tools to maintain the dies at 25 °C bulk temperature for completing every experiment. As to quench the boron steel sheet metal, the hot forming tool was used. The hydraulic press machine was used to apply the pressure to the boron steel sample. The details of the heat transfer experiment performed were explained in this section.

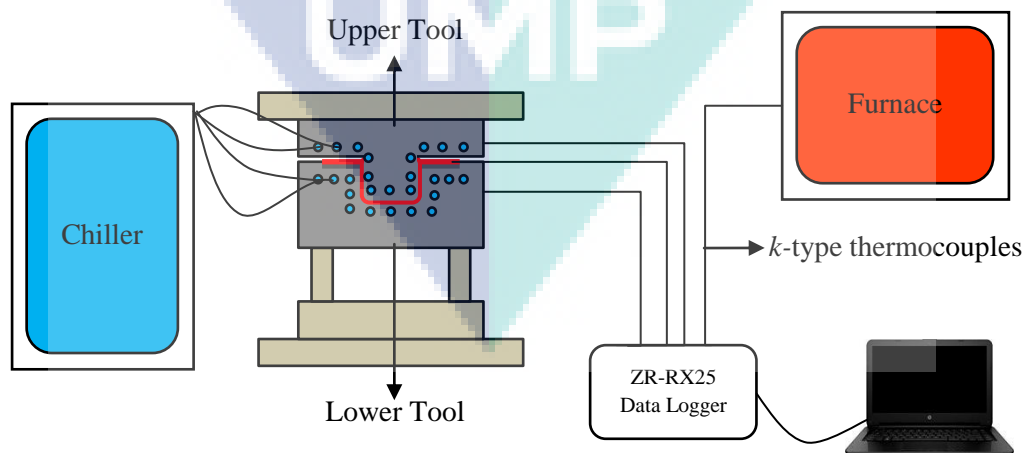


Figure 4.7 The experimental equipment setup for hot press forming tool



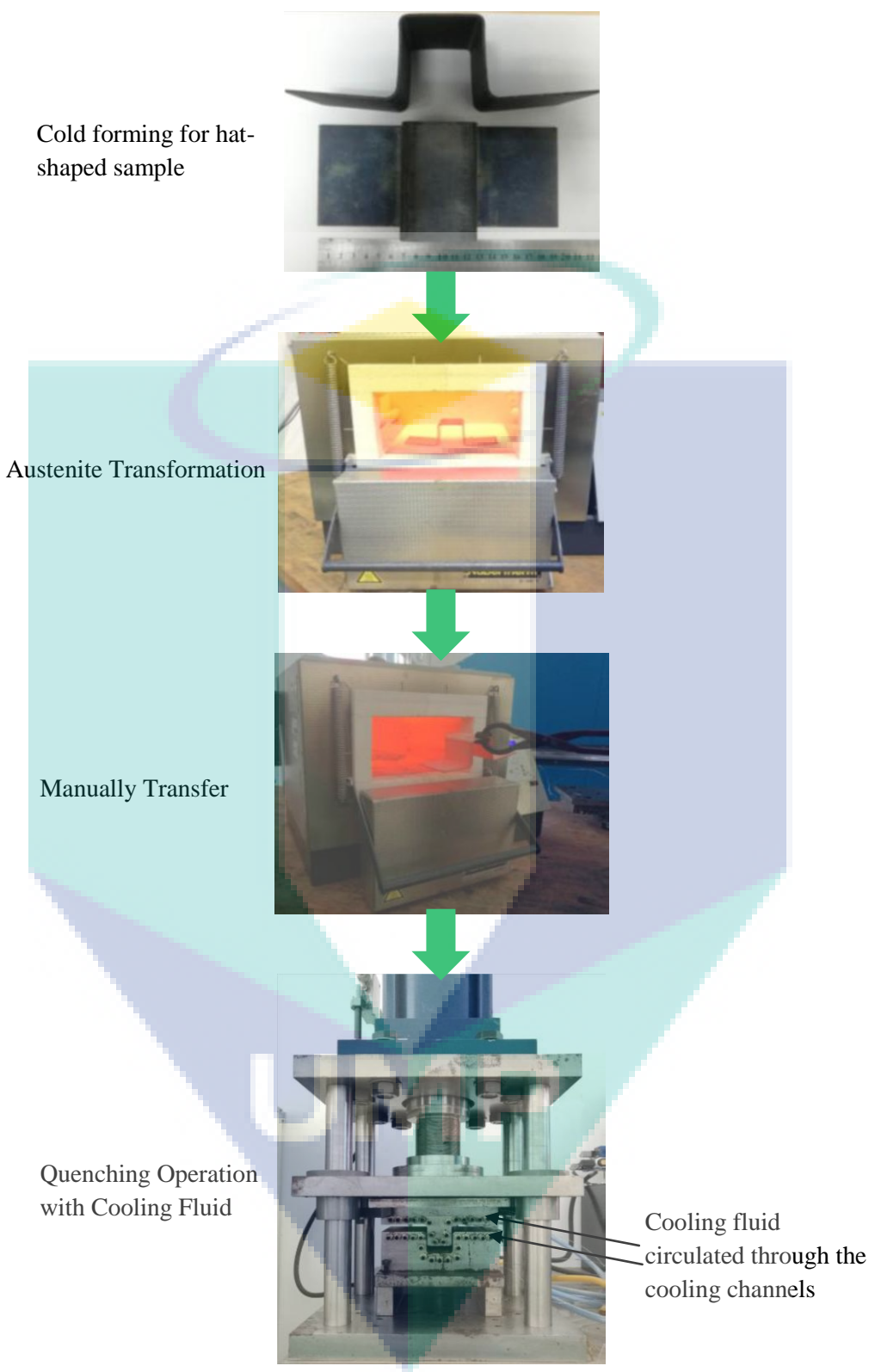


Figure 4.8 Hot press forming process sequence of hat-shaped blank



Prior to HPF experiment, the hat-shape blank was heated to the temperature of 900 °C in the furnace. The austenite blank was then rapidly transferred to the enclosure dies to avoid the heat loss before starting the forming operation. Next, the hat-shaped blank was formed in the HPF dies with a pressure of 20 MPa by using hydraulic press machine as shown above in Figure 4.8. The heat transfer distribution was evaluated during the HPF process. The temperature of the sheet metal blank and the HPF dies were recorded by means of the *k*-type thermocouples. The data collected from experiments were then compared with the simulation results. Figure 4.8 shows the flow of the HPF process of hat-shaped tool. Finally, the hot formed part will be measured and analysed by conducting the hardness measurement, tensile test and metallographic study of the microstructure.

#### **4.4.2 Tensile test measurements**

After hot press forming (HPF) experiments, the hot formed parts were cut into tensile test specimens taken from the wall, bottom and flange locations as illustrated in Figure 4.9. According to the standard of ASTM E 8M, specimens with a total length of 100 mm were cut into 33 mm X 5 mm X 1.8 mm gauge size, which maintained for every tensile test experiment as shown in Figure 4.10. Then, the tensile test specimen was clamped by using the upper and lower jaw of the Universal Tensile Machine (UTM) as depicted in Figure 4.11. Table 4.3 summaries the specifications for UTM machine. The process was stopped when it reached the maximum tensile strength value. The raw results are in the form of force versus elongation. It covers from the elastic phase transition to the plastic deformation until the samples fractures. The data will then be processed to generate the true stress versus true strain curve method regarding ASTM E 8M standard (Merklein et al., 2014; Kim et al., 2015).

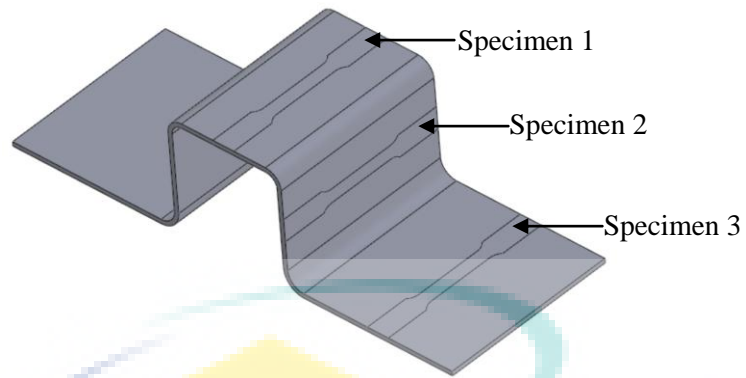


Figure 4.9 Location of hot formed samples for tensile specimen, hardness test and microstructure analysis

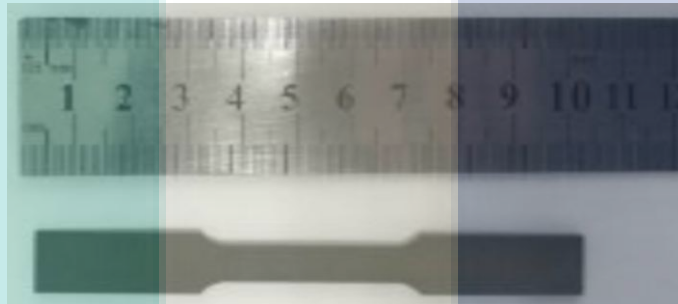


Figure 4.10 Specimen for tensile strength test

Table 4.3 Specifications of Universal Tensile Machine

Parameters	Capacity
Load Capacity (kN)	50
Height (mm)	1582
Width (mm)	756
Depth (mm)	707
Space between Columns (mm)	420
Total Vertical test Space (mm)	1193
Total Crosshead Travel (mm)	1122
Maximum Speed (mm·min <sup>-1</sup> )	500
Minimum Speed (mm·min <sup>-1</sup> )	0.005
Maximum Force at Full Speed (mm·min <sup>-1</sup> )	25
Maximum Force at Full Load (mm·min <sup>-1</sup> )	250
Return Speed (mm·min <sup>-1</sup> )	500
Weight with Typical Load Cell (kg)	141
Maximum Power Requirement (W)	700

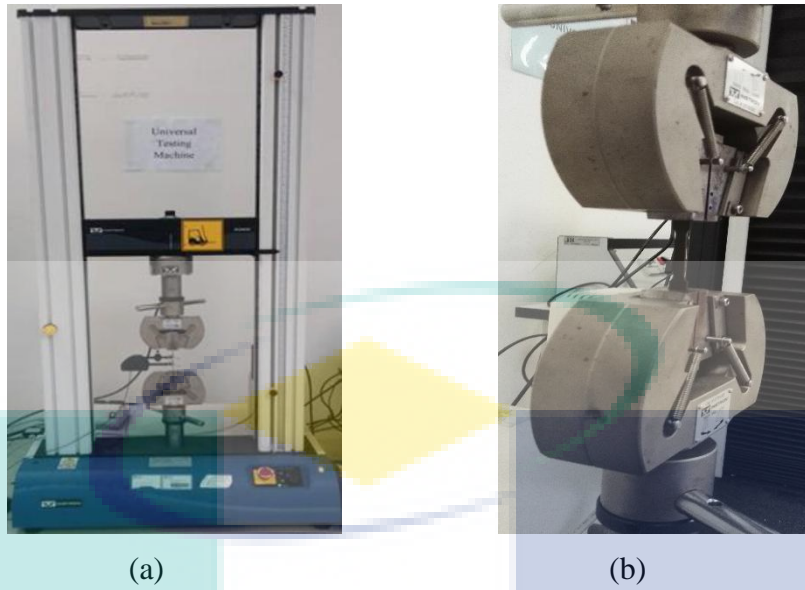


Figure 4.11 Tensile strength measurement with Universal Tensile Machine

#### 4.4.3 Micro-hardness measurements

After the tensile strength test had been done, the sample was then machined into small pieces for the hardness measurement by using Vickers Micro-hardness Machine as shown in Figure 4.12. Table 4.4 lists the specifications of the Vickers Micro-hardness Machine of model Wilson Vickers 402 MVD. Then, the mounting process was followed, where the mixture of 10 ml of resin and 20 ml of powder. The mixture was manually stirred uniformly with a glass rod and held for approximately 3 minutes after the mixture was poured into the reference sample case. Then, the reference sample was subjected to the grinding and polishing operations. Several grades of sand paper from 800-1200 grade grain size and liquid of diamond were used to polish and refine the sample. The hardness value was measured by using Vickers method according to DIN EN ISO 6507-1 with the hardness readings measured by a test force of 1 N (Bardelcik et al., 2012, Maeno et al., 2015, L b be et al., 2016). The diamond shapes for the reference sample was produced on the surface and it was measured by using the Vickers Micro-hardness Tester Machine.

Table 4.4 Specifications of Wilson Vickers 402 MVD machine

Parameters	Capacity
Hardness Scale	Vickers and Knoop
Test Load (kN)	10-2000
Dwell Time (s)	5-99
Eyepiece Magnification	10X
Maximum Specimen Height (mm)	85
x-y Stage Dimension (mm)	100 X 100
x-y Stage Travel Range (mm)	25 X 25
Minimum Reading (mm)	0.01
Operating Temperature (°C)	10-38
Machine Dimension, <i>L x W x H</i> (mm)	513 X 320 X 470
Weight (kg)	36
Power Supply (VAC)	110-220

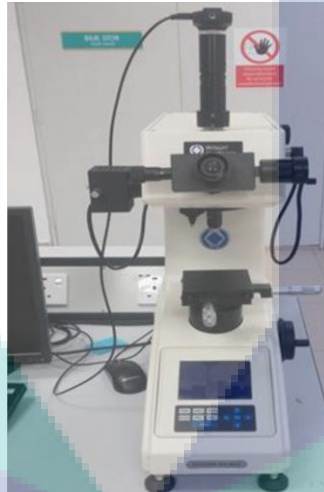


Figure 4.12 Hardness measurement with Vickers Micro-hardness Machine, Wilson Vickers 402 MVD

#### 4.4.4 Metallographic observation study

The obtained microstructures resulting from the samples of hot formed boron steel were analysed by Light Optical Microscopy (LOM) machine with Progress Capture 28.8 Jenoptik Optical System Image Analyser software as shown in Figure 4.13. The inaccuracy of about  $\pm 5\%$  in measuring quantitative area fractions of different phases was reported in the software manual (Nuraini et al., 2013). Table 4.5 lists the specifications of the LOM machine of model Olympus BX51M. The preparation of the reference samples required mounting process as similar to hardness measurement. The

reference sample was then subjected to the mechanical polishing procedures with several grades of sand paper from 100 to 1500 grade grain size and liquid of diamond were used to polish the reference sample. Lastly, the samples were etched with 3% nitric acid for approximately 2 to 5 s time period (Löbbe et al., 2016). The microstructure morphology was observed with a magnification of 20X, 50X and 100X with LOM machine. Then the results of microstructure were captured by using LOM software to generate data and produced the images in the desktop set.

Table 4.5 Specification of LOM, Olympus BX51M machine

Features	Capacity
Optical System	UIS2/UIS (Universal Infinity System) Optical system (featuring infinity correction)
Stroke per Rotation (mm)	0.1 (Fine); 17.8 (Coarse)
Full Stroke Range (mm)	25
Observation Tube Inclination	30°
Distance Adjustment (mm)	50-76
Stage Size, <i>D</i> X <i>W</i> (mm)	156 X 191
Stage Movement, <i>x-y</i> (mm)	76 (Horizontal); 52 (Vertical)
Revolving Nosepiece	Universal 6-position centered revolving nosepiece
Observation Tube	Super widefield trinocular
Magnification Lens	10X-100X (FN 26.5 Super Widefield)
Operating Temperature (°C)	5-40
Operating Environment	Indoor use; Max 2000 m (Altitude)
Power Consumption (W)	140



Figure 4.13 Microstructural analysis by using light optical microscopy machine

#### 4.5 Thermal Analysis of Nanocoolant and Chilled Water in Hot Press Forming

The thermal analysis of hot press forming (HPF) process was conducted by introducing two different cooling agents such as nanocoolant and chilled water separately to compare the heat transfer distribution values. The temperature changed of the heated blank and HPF dies during the process was measured by the temperature sensors as mentioned in Section 4.3 and compared between nanocoolant and chilled water as tabulated in Table 4.6. The cooling rate of nanocoolant was slightly higher than chilled water in experiment. However, both of the coolants have a similar cooling pattern for blank and tool to a certain extent. The pattern agrees well with the trend of cooling performance in HPF process studied by several researchers (Merklein et al., 2008; Mori et al., 2009; Karbasian et al., 2010; Wu et al. 2014).

Table 4.6 Temperature distribution for HPF tools and heated blank

Time, (s)	Nanocoolant			Chilled Water		
	<i>Upper die</i>	<i>Lower die</i>	<i>Blank</i>	<i>Upper die</i>	<i>Lower die</i>	<i>Blank</i>
0.0	25.00	25.00	900.00	61.58	73.81	900.00
0.5	55.00	50.00	537.40	134.64	139.07	637.99
1.0	83.43	83.26	395.91	207.70	204.32	544.32
1.5	110.34	111.00	308.88	200.88	197.19	466.00
2.0	134.74	140.00	266.09	194.06	190.06	375.67
2.5	149.03	149.13	249.37	192.36	186.69	345.40
3.0	160.32	160.00	227.77	190.66	183.32	315.13
3.5	164.20	159.45	206.07	185.63	178.03	292.75
4.0	155.25	152.00	189.97	180.61	172.73	270.37
4.5	146.32	146.10	179.57	174.66	167.28	246.13
5.0	142.72	140.00	166.77	168.70	161.83	221.88
5.5	134.10	133.90	159.67	162.94	156.38	204.65
6.0	127.23	130.03	144.87	157.18	150.93	187.41
6.5	121.31	123.00	139.57	151.98	145.75	174.88
7.0	116.84	116.60	134.57	146.78	140.56	162.35
7.5	109.77	108.50	120.13	142.17	135.76	153.03
8.0	104.50	104.00	112.57	137.56	130.95	143.71
8.5	100.15	99.90	105.98	127.53	124.85	135.61
9.0	94.10	93.78	98.56	117.50	118.75	127.50
9.5	91.94	91.00	96.57	115.06	115.62	123.13
10.0	89.71	89.50	95.33	112.62	112.50	118.75

Figure 4.14 shows the mean and maximum enhancement of nanocoolants cooling rate value compared with the chilled water are 30.2% and 42.3%, respectively. Merklein et al. 2014 compared the three different types of the cooling agents such as pure air, water and atmospheric condition. The results showed that their experimental value obtained a 66.3% augmentation of cooling rate from atmospheric condition to pure air with the controlled air blowing system. Nanocoolants had optimum cooling rate because the convection heat transfer of nanoparticles and Brownian motion in the base fluids (Sundar et al., 2014; Lim et al., 2016). The thermal conductivity of nanocoolant enhancement also depends on the effect of the base fluid (Lim et al., 2016). Nevertheless, chilled water only exhibited convection heat transfer mechanism with the cooling rate around  $50 \text{ K}\cdot\text{s}^{-1}$  (Karbasian et al., 2010).

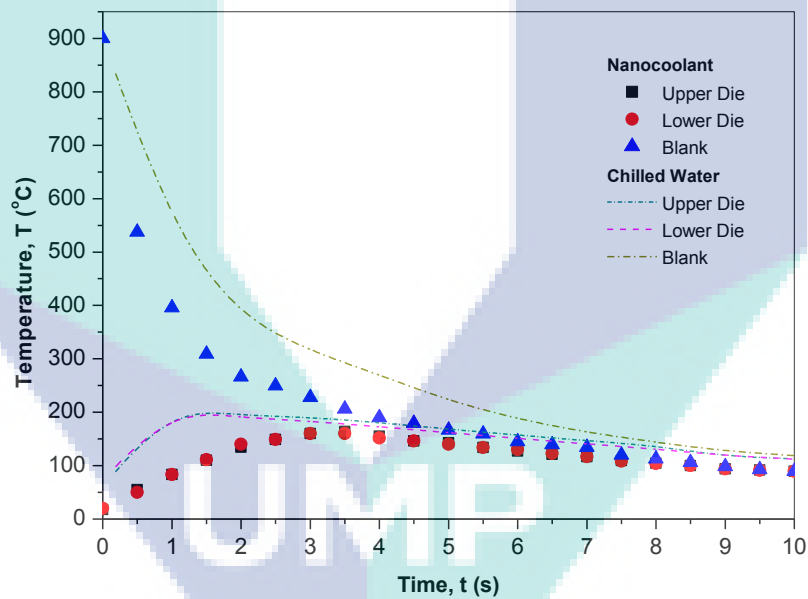


Figure 4.14 Heat transfer distribution between nanocoolant and chilled water for hat-shaped tool



#### 4.6 Microstructural Transformation Analysis of Hot Pressed Boron Steel

When the austenitization was applied on boron steel, higher temperature for homogenization was mandatory. Since the transformation was strongly dependent on the microstructure in terms of the chemical composition, the present phases and the grain size (Lolla et al., 2011). The micrographs of as-received and hot pressed boron steel blanks were tabulated according to the period of holding time or dwell time taken and types of cooling fluids as shown in Table 4.7. At 100X magnification, the as-received sample contained the mixture of pearlite phase and 73-77% ferrite and a small amount of carbide. Nuraini et al. (2013) stated that as-received boron steel exhibited pearlite phase located at ferrite grain boundaries. In this phase, ferrite-pearlite microstructure existed and brittle fracture in the sheet metal steel in the state (Frydman et al., 2012). For hot pressed samples at 100X magnification, they showed the martensitic microstructure and grain refinement occurred in the blank samples.

After hot forming and nanocoolant quenched for 3 s of holding time, the sample contained pearlite and martensitic microstructure. The pearlite content was around 2%, and the martensite content was approximately 90%. Srithananan et al. (2016) stated that brown colour represented martensitic phase after the image was taken to quantify the area fraction. The figure indicated that short holding time was insufficient to enable a martensitic transformation. Löbbe et al. (2016) demonstrated an experiment to study the first phase transformation of boron steel at each dwell time and cooling rate. They found that longer holding time and a cooling rate above the critical cooling rate of  $45 \text{ K's}^{-1}$  were feasible to reach the martensite start and develop a martensitic microstructure, which was determined and agreed by Naderi et al. (2011).

Table 4.7 Micrographs of boron steel blank with several quenching time periods

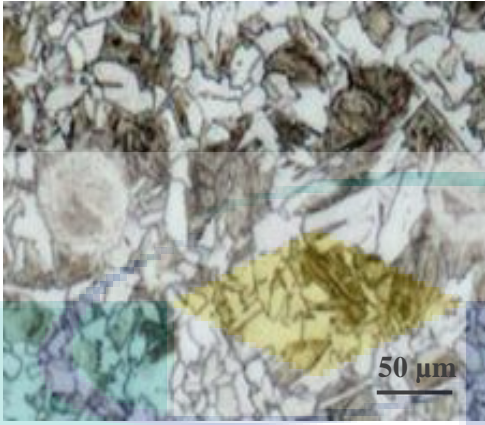
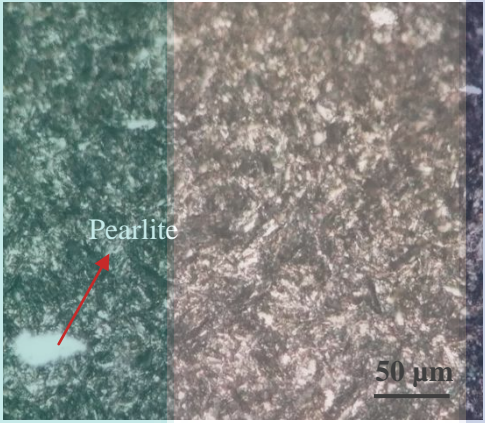
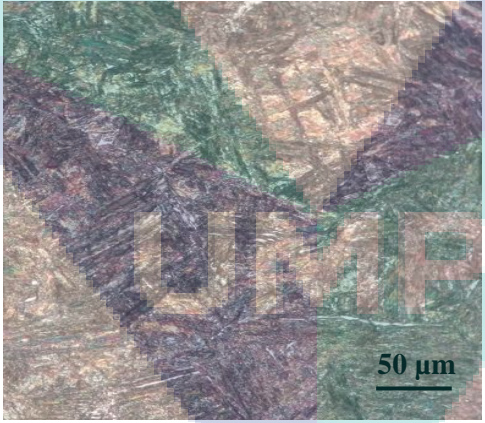
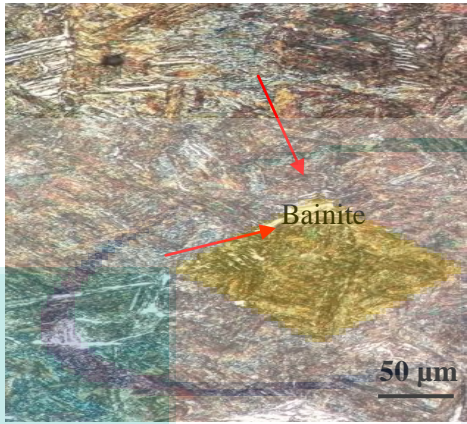
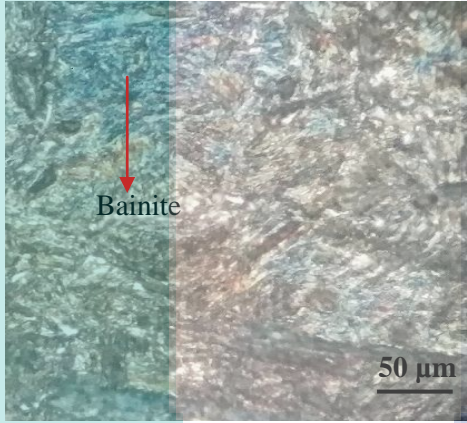
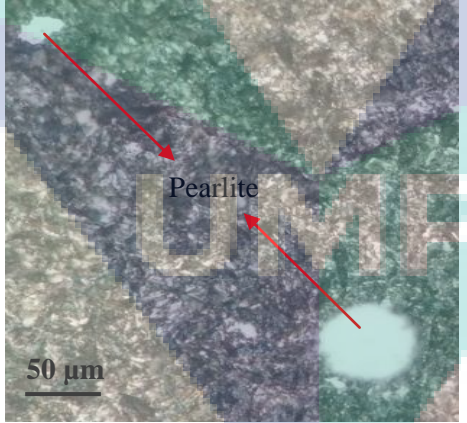
Cooling Fluid	Microstructure	Evaluation
As-received		<p>Material : Boron steel                      Microstructure : Ferrite and Pearlite</p>
Nanocoolant		<p>Material : Boron steel                      Temperature : 900 °C                      Holding Time : 3 s                      Microstructure : Pearlite and Martensite</p>
Nanocoolant		<p>Material : Boron steel                      Temperature : 900 °C                      Holding Time : 5 s                      Microstructure : Martensite</p>

Table 4.8 Micrographs of boron steel blank with several quenching time periods  
(Continued)

Cooling Fluid	Microstructure	Evaluation
Nanocoolant		Material : Boron steel Temperature : 900 °C Holding Time : 8 s Microstructure : Martensite and Bainite
Nanocoolant		Material : Boron steel Temperature : 900 °C Holding Time : 10 s Microstructure : Bainite and Martensite
Chilled Water		Material : Boron steel Temperature : 900 °C Holding Time : 10 s Microstructure : Pearlite and Martensite

In contrast to the 3 s of holding time, the sample of nanocoolant quenched for 5 s of dwell time contained a nearly total martensitic microstructure. The martensite content was more than 97% and only approximately 0.01% of bainite content in the transformation phase. The microstructure result showed that the nanocoolant with high

cooling rate used was sufficient for 5 s quenching time to produce a fully martensitic microstructure with large needles shaped martensite were oriented in different angles and observed in almost every part of the sample, which suggested an increase of strength with sufficient cooling rate and holding times. This result was in accordance with Nikravesh et al. (2012) and LÖbbe et al. (2016), who showed that higher cooling rate and sufficient or long holding times cause a reduction of the martensite start temperature. Sufficient dwell time caused a coarser grain structure led to a formation of martensite than a diffusion controlled formation of the cementite because of the increasing diffusion length (Naderi et al., 2011). This mechanism finally required a higher driving force for the martensite transformation, which was obtained at a lower temperature of hot forming tools with a cooling fluid of high convection heat transfer coefficient (LÖbbe et al., 2016).

Furthermore, the phase transformation of hot formed boron steel by 8 s and 10 s quenching period of time as shown in figures. The results presented that the microstructure exhibited a slight content of bainite microstructure, which blue colour represented bainitic phase (Srithananan et al., 2016) after 8 s of quenching time. The martensite content was approximately 95%, while bainite content was around 3%. However, in 10 s quenching period of times, the hot pressed sample exhibited an increasing content of bainite microstructure. The bainite content was more than 15% and the martensite content decreased to only 74%. The reason for longer holding times promoted high bainitic fractions, because of the martensite transformations were impoverished of carbon so that a recombination of martensite was impeded (Bargel et al., 2012). LÖbbe et al., (2016) stated that the higher temperature and longer holding time caused a grain growth of hot formed sample. Thus, the diffusion of carbon was reduced and fewer lattice defects were available for the nucleation of carbides so that the diffusionless transformation of ferrite and bainite was favoured. Obviously, the amount of martensite significantly decreased when the applied quenching time was increased, while the amount of bainite increased in contrast.

Lastly, the cooling rate of chilled water was not sufficient to transform the hot pressed sample to a fully martensitic microstructure. Two phases of the pearlite and mixture of bainitic/martensitic microstructures obtained by lower cooling rate was anticipated. Nevertheless, with the realized higher cooling rate from the austenitization



temperature in these heat treatments, ferrite and pearlite could be prevented (Bardelcik et al., 2014). After hot forming and water quenched in 10 s period of holding time, the sample contained the mixture of pearlite phase and bainitic/martensitic microstructure. The pearlite content was around 5%, the bainite content was more than 40% and the martensite content was decreased to approximately 50%. LÖbbe et al., (2016) concluded that the reason for the cooling rate-dependent phase transformation was an inhomogeneous carbon distribution, so that a critical concentration allowed the diffusion control transformation. Hence, the critical cooling rate was the major controlling factor of martensite formation but rather increasing of short holding time and temperature.

#### **4.7 Tensile Strength Analysis of Hot Pressed Boron Steel**

The experimental results obtained from the tensile strength test was tabulated according to the periods of quenching time taken and types of cooling fluids as shown in Table 4.9. The graph reflected the data recorded in Table 4.9 were plotted accordingly as displayed in Figure 4.15. The figure shows the ultimate tensile strength of hot pressed samples in two different cooling mediums such as nanocoolant and chilled water with several quenching durations.

Since a nearly total martensitic microstructure was obtained at 5 s period of holding time with nanocoolant quenched, the maximum tensile strength 1521.93 MPa was attained with higher convection heat transfer coefficient and cooling rate at sufficient period of holding time which was 5 s quenching duration. This effect was driven by the large martensitic needles oriented in different angles, which increased for higher convection heat transfer coefficient in order to increase the cooling rate and not more than 5 s holding duration. Bardelcik et al. (2010) experimentally investigated the effect of cooling rate on the high strain rate properties of boron steel and they proposed that the cooling rate was accelerated by increasing the convection heat transfer coefficient, more nucleation points were utilized and hence, a finer and disordered martensitic microstructure was formed. However, the tensile strength value of the boron steel was decreased gradually when increased the quenching time. According to LÖbbe et al. (2016) for setting mechanical properties of high strength steels in rapid hot

forming processes, the ultimate tensile strength of boron steel was decreased gradually when the dwell time or dies holding time increased. This phenomenon could be explained by the producing lath-shaped structure because a finer structure and grain boundaries impede dislocation motions.

Table 4.9 Tensile strength value for several specimens of hot pressed boron steel

Cooling Fluids	Quenching Duration (s)	Ultimate Tensile Strength (MPa)		
		Specimen 1	Specimen 2	Specimen 3
Nanocoalant	3	1282.67	1340.46	1295.35
	5	1500.08	1521.93	1501.79
	8	1421.66	1419.84	1440.55
	10	1378.11	1396.21	1451.62
Chilled Water	10	1110.67	1030.55	1104.65
As-received	0	545.28	541.32	523.67

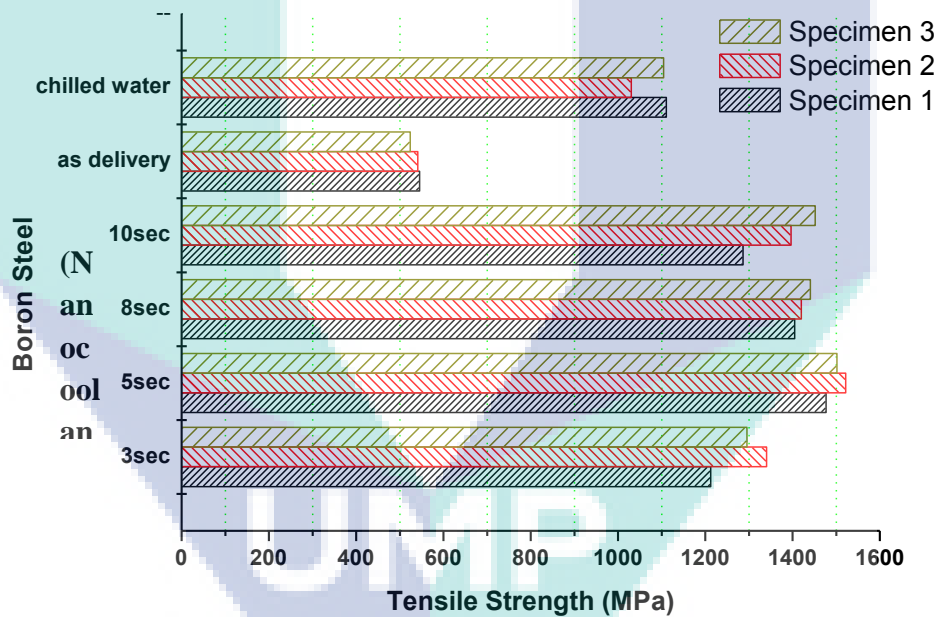


Figure 4.15 Ultimate tensile strength of blank samples at several cooling conditions

On the other hands, the ultimate tensile strength of hot pressed boron steel with chilled water quenched was only 1030.55 MPa. Since the micrograph of sample enforced a slight pearlite fraction and mixture phase of bainitic/martensitic microstructures. It was not only the corresponding tensile strength which was unfavourably low but also the small uniform strain indicated a brittle microstructure (Löbbeck et al., 2016). The comparison of the tensile strength of the hot pressed boron

steel with nanocoolant quenched and the hot pressed boron steel with chilled water quenched and the as-received boron steel before the quenching process. The tensile strength increased with the increasing of convection heat transfer coefficient in order to increase the cooling rate. The ultimate tensile strength enhancement ratio was 47.5% with nanocoolant quenched compared with the chilled water quenched of the hot pressed boron steel. Furthermore, the tensile strength of boron steel as received was only 545.28 MPa. The tensile strength of hot pressed boron steel with nanocoolant quenched had increased up to 179.1% enhancement when compared with the raw material.

According to Merklein et al. (2008), the value of tensile strength after quenching process was around 1400 MPa due to the specimen showed an increase of the martensitic microstructure. Srithananan et al. (2016) experimental study the stress-strain and fracture behaviour of heat-treated boron steels for HPF process and they stated that stress-strain curves of the heat-treated boron steels were strongly influenced by the occurring microstructure constituents. The tensile strength of boron steel as received stage was low due to the bigger portions of ferrite phase microstructure were detected (Naderi et al., 2011). However, the ultimate tensile strength value variations of the investigated hot formed specimens had been anticipated. Naderi et al. (2011) presented that a decreased in tensile strength value, because of the martensite volume fraction reduction. The martensitic phase microstructure due to its high hardness was brittle and low ductility. Moreover, Wang et al. (2013) conducted an investigation of the die quench properties of hot forming, and they found that if the transferring time of austenite boron steel to the hot forming tools more than 15 s could affect the strength of hot pressed boron steel around 400 MPa reduction. Therefore, in this research project, hot pressed boron steel with nanocoolant quenched in 5 s die holding duration would increase the ultimate tensile strength up to 2000 MPa by using robotic arm transferred the austenite boron steel.

#### **4.8 Hardness Analysis of Hot Pressed Boron Steel**

The Vickers hardness results of the hot formed boron steel in several periods of quenching time taken and types of cooling fluids were displayed in Table 4.10. The

hardness values were measured by using Vickers method according to DIN EN ISO 6507-1 with the diamond shaped was produced on the surface of the sample as depicted in Figure 4.16. The graph reflected the data recorded in Table 4.10 were plotted accordingly as shown in Figure 4.17. The figure shows the hardness value of as-received boron steel and hot pressed samples in two different cooling fluids such as nanocoolant and the chilled water.

Table 4.10 Hardness value for several specimens of hat-shaped boron steel

Cooling Mediums	Quenching Duration (s)	Vickers Hardness (HV1)		
		Specimen 1	Specimen 2	Specimen 3
Nanocoolant	3	491	421	436
	5	588	543	552
	8	579	526	520
	10	570	540	549
Chilled Water	10	567	578	509
As-received	0	105	106	105

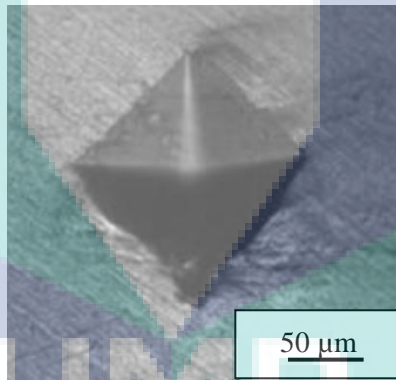


Figure 4.16 Vickers hardness reading HV20 with diamond shaped indent 50 μm

Hardness properties of the hot pressed boron steels as different quenching mediums and quenching duration were responding to micrograph as shown in Table 4.7. The highest Vickers hardness value of 588 HV1 was measured in nanocoolant with the quenching time period of 5 s. Note that the as-delivered boron steel had the average micro-hardness of 105 HV1. The quenching in the nanocoolant led to significant increase in ratio of 4.6 times in hardness of the sample due to the presence of large martensitic needles structure in the entire region (Namklang et al., 2016). The



martensitic structure would lead to an elevated strength of the boron steel due to an increase in dislocation impedances (Bardelcik et al., 2010 & Wang et al., 2007).

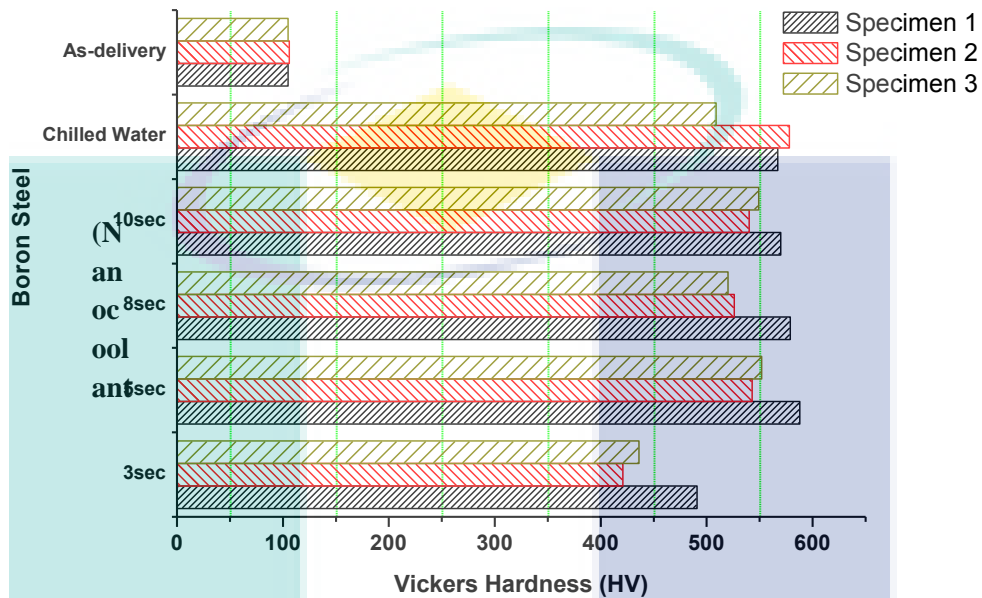


Figure 4.17 Hardness value of hot pressed samples at several cooling conditions

On the other hand, hot pressed samples with the presence of bainitic microstructure phase exhibited a marginal increase in the hardness value. The lowest hardness was only 421 HV1 among these samples, when hot pressed in chilled water and long period of quenching time. Eller et al. (2014) highlighted that as the hardness value was higher than 470 HV or 47.5 HRC, then it could be identified as a resulting of martensitic microstructure phase. As the hardness value was below 450 HV, then it could be related to ferrite/pearlite mixed phase, and it was expected that some pearlite zone had been formed in the microstructure of hot pressed sample with 3 s quenching time period. It was in agreement with the results presented in Table 4.10. Merklein et al. (2008) found that the hardness value of hot pressed part was around 514 HV. Naderi et al. (2011) obtained the highest hardness value of approximately 600 HV in their sample of steel-A with different carbon contents. Namklang et al., 2016 produced the hot pressed part with Vickers hardness value of 550 HV in the bottom area region. Since, the hardness value obtained was higher compared to the normal trend, the results were acceptable.

## 4.9 Summary

This chapter outlines the design experimental analysis and investigation of hot press forming (HPF) process in order to achieve the objectives. This research has begun with the hot forming tool setup with *k*-type thermocouples before the preparation of hat-shaped samples and nanocoolant for the experimental HPF process that have been explained in Section 4.2.2 and 4.2.3. Next, the experiment of HPF process by using boron steel was conducted involving several cooling fluids with different cooling rates experiment, heat transfer experiment, microstructure morphology observation by Light Optical Microscope, and the tensile strength test measurement by using Universal Tensile Machine, and the Vickers hardness measurement by using Vickers Microhardness Machine. The results were presented and discussed briefly in the last few sections of Chapter 4.

In this study, thermal analysis was conducted for the heated blank, upper die and lower die of the HPF tool. This approach was able to measure the heat transfer distribution of the austenite blank and hot forming tool by introducing nanocoolant and chilled water into the cooling channel system. The experimental results attained demonstrate an acceptable agreement with Namklang et al. (2016). The metallographic studies of the hot pressed parts were performed by using Light Optical Microscope (LOM) machine with Progress Capture 28.8 Jenoptik Optical System Image Analyser software. The microstructure transformation of the hot pressed part with 5 s quenching time by using nanocoolant exhibited nearly total martensitic phase. The value of the tensile strength and Vickers hardness were also measured. The tensile strength of the final part was observed to increase up to 47.5% with nanocoolant quenched compared with the chilled water, and approximately 179.1% enhancement in tensile strength when compared with the as-received boron steel. The enhancement ratio of hardness was about 4.6 times for the hot pressed part compared with the initial condition of boron steel. As a concluding remark, it was apparent that the nanocoolant which obtained higher heat transfer coefficient in order to produce higher cooling rate compared with chilled water were able to achieve better mechanical properties such as tensile strength and hardness values of the output sample.

## CHAPTER 5

### CONCLUSION

#### 5.1 Conclusion

The overall objectives of the research project were achieved in which the potential of the selected aluminium oxide,  $\text{Al}_2\text{O}_3$  nanoparticles suspended in aqueous ethylene glycol solution of base fluid has been investigated through a series of methods and analyses. The three research objectives were achieved through three different stages of experiments and summarised into three different paragraphs.

The analyses in the first stage of the experiment were conducted to fulfil the objective one. From this first stage of the experiment, the nanocoolants produced from nanoparticles of  $\text{Al}_2\text{O}_3$  was uniformly dispersed in water-ethylene glycol mixture could sustain in sedimentation evaluation after being synthesized by using the ultrasonic hot-air machine. Besides that, increasing of nanoparticle concentrations and a reduction of ethylene glycol base fluids was proven to enhance thermal conductivity property in order to increase the convection heat transfer coefficient value. The enhancement of heat transfer coefficient could attain 66.2% compared with chilled water as a cooling agent in the HPF process at the bulk temperature condition of 25 °C. Also, the results showed that the several volume fractions of  $\text{Al}_2\text{O}_3$  nanoparticles and different concentrations of ethylene glycol as base fluids owned their respective enhancement and weaknesses of convection heat transfer for application in the cooling system of HPF process. Therefore, finding the ideal volume fraction of  $\text{Al}_2\text{O}_3$  base water-ethylene glycol mixture nanocoolant is important to increase the productivity of HPF processes. The use of  $\text{Al}_2\text{O}_3$ /water-ethylene glycol mixture nanocoolant of 60%:40% (W/EG) with

a volume concentration of 0.8% is recommended as cooling nanofluids for application in HPF process.

Through the second stage of thermal analysis, the second objective was achieved. Thermal analysis in the HPF process of heat transfer distribution for the upper tool, lower tool and heated blank were conducted in HPF experiment with nanocoolants and chilled water. The temperature changed of hat-shaped tools and heated blank were analysed with respect to the several periods of quenching time. It was found that the results obtained at this stage corresponded well with those stated in the literature and hypothesis in which nanocoolants of  $\text{Al}_2\text{O}_3$  base fluid could help to rapidly reduce the temperature of tools and heated sheet metal blank by improving the convection heat transfer coefficient value to increase the cooling rate. Also, the optimum quenching time and heat transfer enhancement of nanocoolant compared with chilled water were collected from the analysis. As for the validation of experimental results with other reviews, the percentage deviations of heat distribution for the upper tool, lower tool and heated blank were able to fulfil the requirements and agreed with the experimental hat-shaped.

The analyses in the final stage of the experiment were conducted to achieve objective three. From this final stage of the experiment, the tensile strength of test specimens produced from three different locations such as flange, wall and bottom of the austenite boron steel blank after being manufactured from wire cut machine. Moreover, the specimen was cut into a small piece of samples after the tensile strength test had been performed. The samples were mounted for Vickers hardness measurement after the grinding and polishing operations had been performed for the samples. Also, the microstructure analysis of the samples was conducted to evaluate the result of heated boron steel from austenite phase transformed to fully martensitic behaviour. The result showed that the mechanical properties of hot formed boron steel were improved by introducing nanocoolant compared with chilled water to a certain extent. The nanocoolant as a cooling agent for HPF experiment obtained fully martensitic microstructure of boron steel and thus contributing to the higher tensile strength and Vickers hardness value compared with boron steel as received condition. Chilled water for HPF experiment, on the other hand, showed the pearlite microstructure transformation due to the lower convection heat transfer value and cooling rate.

Therefore, the tensile strength of hot formed boron steel was able to fulfil the minimum requirement of HPF value with 1400 MPa, but the hardness value was still in acceptance limit which was above 470 HV. In addition, the strength of boron steel from nanocoolants was evaluated to increase up to 190.90% after the quenching operation, while the hardness was approximately enhanced 414.28% from the as delivered condition. As compared with chilled water, the tensile strength was approximately improved 47.5%, but the Vickers hardness value was reminded satisfactory limit rate as hot formed part. The value of the tensile strength measured would be slightly higher than 1600MPa from the usual trend. This was mainly due to the longer transfer time of the blank product from the furnace to the hydraulic press machine, in addition to the waiting time for the machine to press the heated boron steel.

## **5.2 Contributions to Knowledge**

The significant contributions of this research study were to synthesise the nanocoolants as alternative cooling fluid for hot press forming (HPF) applications and optimised the cooling system to develop a high productivity HPF system for automotive industry market in ASEAN region countries. In addition to the nanocoolants application, this research also studied the heat transfer distribution in order to optimise the power consumption by the chiller. The mechanical properties enhancement of hot formed parts was investigated and analysed in order to introduce to the commercial car chassis. This summarises the four original contributions of this research project which are as follows:

- The effect of several concentrations of nanocoolant on the convection heat transfer coefficient with optimization the thermal physical properties in terms of thermal conductivity and dynamic viscosity had been analysed and consequently, the augmentation of heat transfer coefficient value and optimum concentration of nanocoolant could be selected.
- The effect of nanocoolants and chilled water for the hot pressed part on the mechanical properties in terms of tensile strength and Vickers

hardness; and microstructure transformation of several periods of quenching time.

- The successful study of this research could lead to better understanding and optimized the HPF process. The knowledge and data defined for the characteristic of ultra-high strength steel (UHSS) and nanocoolants could be widely used to gain its best performance in the hot forming operation, improved cycle time of production and die life duration.

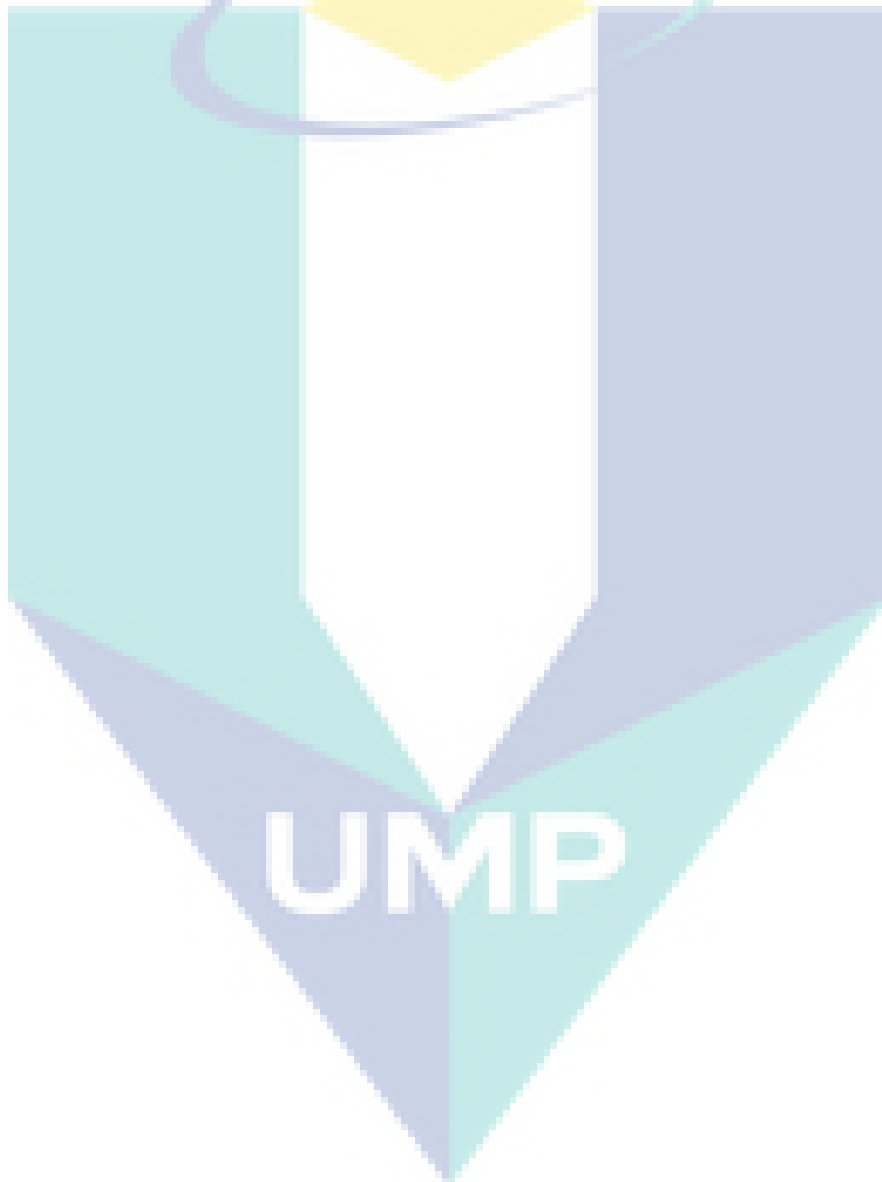
### 5.3 Future Works

Several suggestions are proposed as follows for future work:

- 1) Similar methodologies adopted in this research could be referred and applied for the optimization design of cooling channels. Due to the availability of advanced engineering methods such as Heuristic method, Taguchi method or Neural Networks, the cooling rate of hot press forming (HPF) tools and austenite boron steel blank might be increased during the quenching process. Therefore, the research could be conducted to study the optimization of cooling channel design for the HPF tool process. The results might be improved with nanocoolants compared to the conventional process.
- 2) The automated robotic arm replaced the manually transfer method to transfer the heated blank from the heating furnace to the HPF tools. This robotic arm could be used to reduce the temperature of heated blank lost through atmosphere condition by rapidly transferring the heated blank. Instead of the very slow press speed of hydraulic press machine, this might result in a cooler blank before the quenching operation transpires.
- 3) In order to ensure a sustainable and continuous production of hot formed parts, the directly HPF method could be conducted to eliminate the transfer time of the heated blank. In this process, the blank was formed and hardened in one mold which saved the cost of preforming and accelerated the pace of production. Therefore, research could be conducted to study the thermal-mechanical properties of the sheet metal blank at optimum temperature condition. The mechanical property results of sheet metal blank

might be different owing to the several austenitic temperature conditions and no heat loss in transferring operation.

4) The data from this study have a novelty because there is no report of implementing nanocoolants as the cooling agent for hot forming tools. The study could be published in journal or article as a reference for other researchers who are interested in exploring about ultra-high strength steel (UHSS) materials.





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**APPENDIX A**  
**G-CODE FOR CUTTING TENSILE TEST SPECIMEN FROM HAT-SHAPED PART**

(= ON OFF IP HRP MAO SV V SF C PIK CTRL WK WT WS WP PC SK);  
C000 = 005 014 2215 000 240 +040.0 8.0 0102 0 000 0000 020 120 100 045 0000 00;  
C001 = 006 014 2215 000 251 +030.0 8.0 0102 0 000 0000 020 120 100 045 0000 00;  
H000 = +000000.0100 ;  
H001 = +000000.1470 ;  
( FIG-1 1T ALL CIRCUMFERENCE);  
G54;  
G90;  
G92X-3.0Y1.0Z0;  
G29;  
T94;  
T84;  
C000;  
G42H000G01X-1.0Y1.0;  
C001X0.0;  
H001;  
M98P0001;  
T85;  
G149G249;  
M02;  
N0001;  
G01X0.0Y30.0;  
G03X2.0Y34.4721I-4.0J4.4721;  
G01Y65.5279;  
G03X0.0Y70.0I-6.0J0.0;  
G01Y100.0;  
X10.0;  
Y70.0;  
G03X8.0Y65.5279I4.0J-4.4721;  
G01Y34.4721;  
G03X10.0Y30.0I6.0J0.0;  
G01Y0.0;  
X0.0;  
Y1.0;  
G40H000X-3.0;  
M99;

**APPENDIX B**  
**HEAT TRANSFER COEFFICIENT VALUES OF NANOCOOLANT**

$$\begin{aligned}\text{Flow rate, } \dot{V} &= \left(\frac{l}{\text{min}}\right) = 14 \\ &= \left(\frac{14l}{60s}\right) = 0.2328 \frac{l}{s} \\ &= \left(\frac{m^3}{s}\right) = 2.328 \times 10^{-4} \frac{m^3}{s}\end{aligned}$$

$$\begin{aligned}\text{Volume flow rate, } V &= \frac{\dot{V}}{A} \\ &= \frac{2.328 \times 10^{-4}}{5.027 \times 10^{-5}} = 4.631 \frac{m}{s}\end{aligned}$$

$$\begin{aligned}\text{Reynolds number, } Re &= \frac{\rho_{nf}VD}{\mu_{nf}} \\ &= \frac{(1082.27)(4.631)(0.008)}{0.00401} = 9998.987\end{aligned}$$

$$\begin{aligned}\text{Prandtl number, } Pr &= \frac{C_p \mu}{k_{nf}} \\ &= \frac{3465.46 \times 0.004}{0.425} = 32.698\end{aligned}$$

$$\begin{aligned}\text{Nusselt number, } Nu_{nf} &= 0.025 \times Re_{nf}^{0.76} \times Pr_{nf}^{0.45} \left[ (0.01 + BR)^{-0.1} \left( 1 + \frac{T_b}{70} \right)^{0.14} \right] \\ &= 0.025(9998.987)^{0.76} (32.698)^{0.45} \left[ (0.01 + 0.60)^{-0.1} \left( 1 + \frac{25}{70} \right)^{0.14} \right] \\ &= 189.519\end{aligned}$$

$$\begin{aligned}\text{Heat transfer coefficient, } h_{conv} &= \frac{Nu(k_{nf})}{D} \\ &= \frac{189.519(0.425)}{0.008} = 10068.181 \frac{W}{m^2 K}\end{aligned}$$

**APPENDIX B1**  
**SAMPLE OF COOLING RATE FOR SHEET METAL BLANK**

**Heat transfer of boron steel**

Heat transfer,  $Q = \Delta U = mC_p(T_2 - T_1)$

Where,  $m = 3.294 \times 10^{-2} \text{ kg}$

$C_p = 590 \text{ J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$

Hence,  $Q = (3.294 \times 10^{-2}) \cdot (590) \cdot (375.67 - 162.35)$

$= 4145.789 \text{ J}$

**Cooling rate**

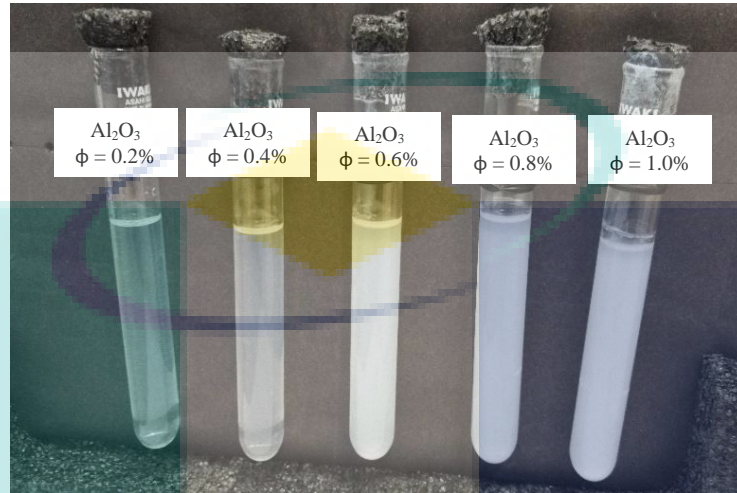
Cooling rate,  $\dot{Q} = \frac{Q}{\Delta t}$

$= \frac{4145.789}{7-1}$

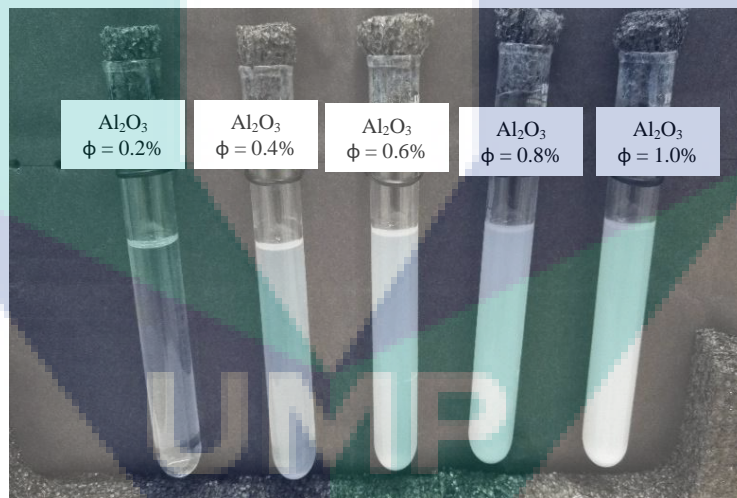
$= 592.256 \text{ J} \cdot \text{s}^{-1}$

**UMP**

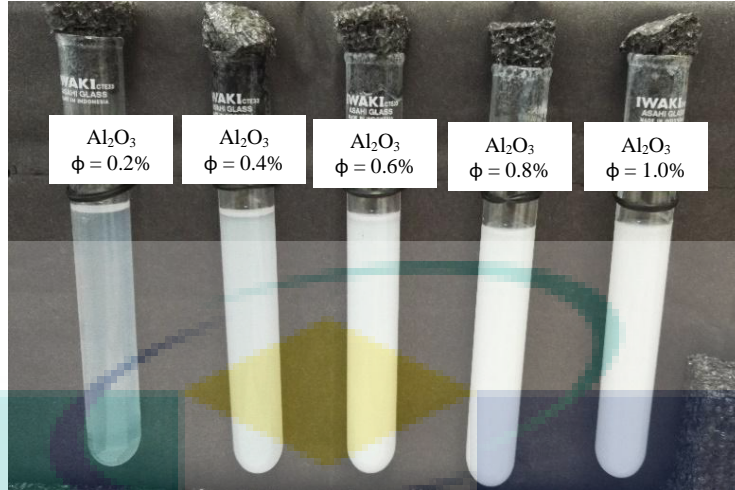
**APPENDIX C**  
**SEDIMENTATION OBSERVATION OF ALUMINIUM OXIDE/WATER-EG**  
**MIXTURE AFTER A MONTH OF PREPARATION**



(a) Water-Ethylene Glycol (60%:40%)



(b) Water-Ethylene Glycol (50%:50%)



(c) Water-Ethylene Glycol (40%:60%)

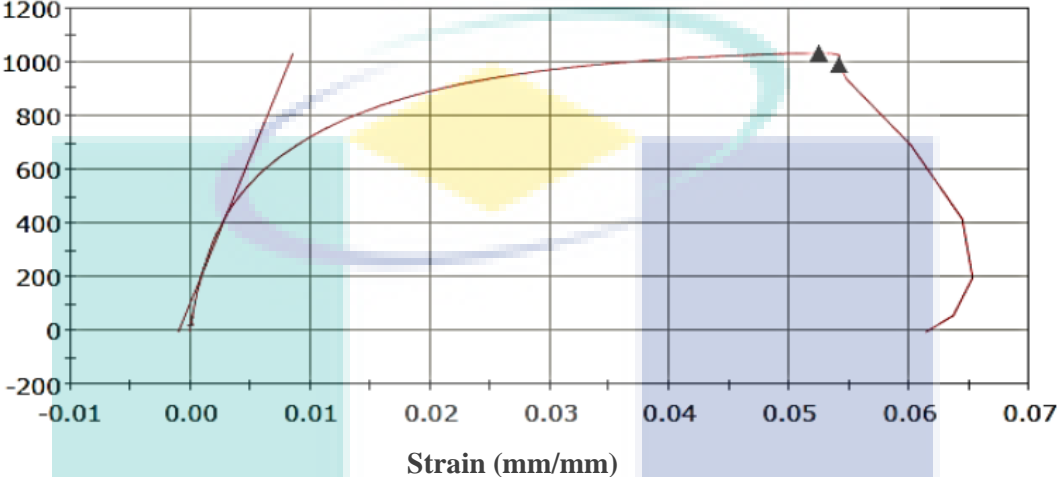
UMP



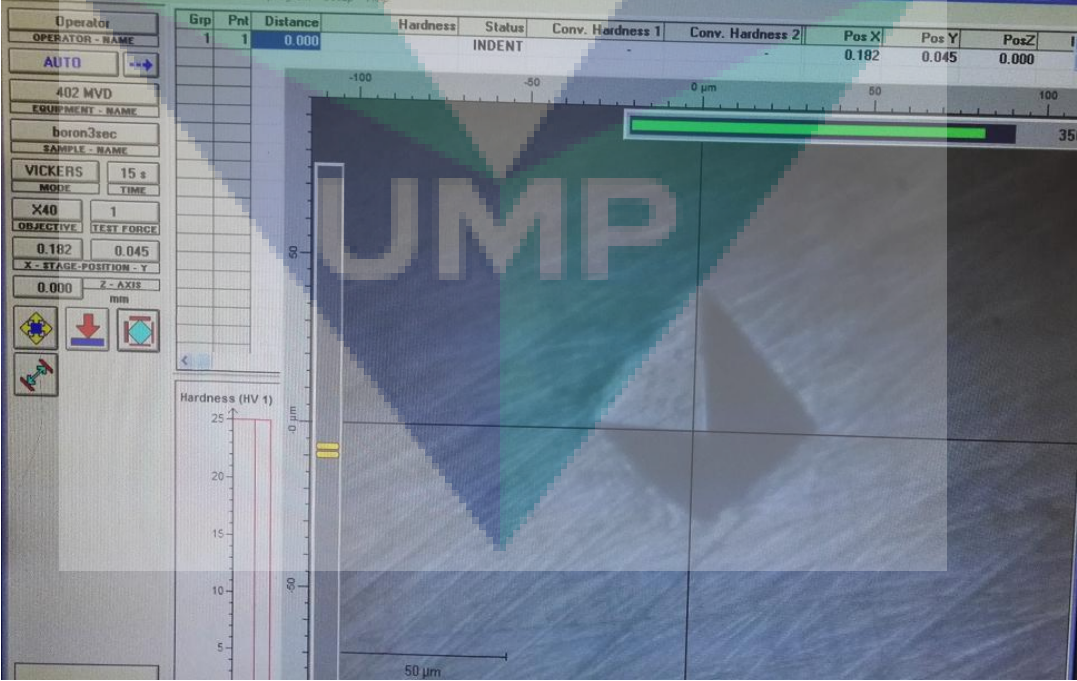
**APPENDIX D**  
**SAMPLE OF TENSILE STRENGTH AND HARDNESS MEASUREMENT**

a)

Tensile stress (MPa)



b)



## APPENDIX E

### INTERNATIONAL COMMUNICATIONS IN HEAT AND MASS TRANSFER

Investigation of thermal conductivity and viscosity of Al<sub>2</sub>O<sub>3</sub>/water–ethylene glycol mixture nanocoolant for cooling channel of hot-press forming die application

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#### ABSTRACT

Hot-press forming process is widely used to produce lightweight chassis in automotive industries. The hot-press forming process currently uses water as coolant to quench boron steels in a closed die with a cooling channel. However, to enhance performance of hot-press forming die, the fluid with better thermal properties will be used instead of normal water. This study dispersed Al<sub>2</sub>O<sub>3</sub> nanoparticles with an average diameter of 13 nm in three volume percentages base ratios of water (W) to ethylene glycol (EG) (i.e. 60:40, 50:50, and 40:60) by two-step preparation. The two main parameters in cooling rate performance are thermal conductivity and viscosity. The nanocoolant of Al<sub>2</sub>O<sub>3</sub>/water–ethylene glycol mixture is prepared for the volume concentration range of 0.2 to 1.0%. The thermal conductivity and viscosity are then measured at temperature range of 15 to 55 °C. The highest enhancement of thermal conductivity was observed to be 10% higher than base fluid for 1.0% volume concentration at 55 °C in 60:40 (W/EG). However, the highest enhancement of viscosity was measured to be 39% for 1.0% volume concentration in 40:60 (W/EG) at 25 °C. The convective heat transfer coefficient of 1.0% concentration in 60:40 (W:EG) at 25 °C is enhanced by 25.4% better than that of 50:50 and 40:60 (W:EG) base fluid. Therefore, this study recommends the use of Al<sub>2</sub>O<sub>3</sub> in 60:40 (W:EG) mixture with volume concentration of less than 1.0% for application in cooling channel of hot-press forming die. Nanocoolant as cooling agent with higher heat transfer coefficient compared to the base fluid can reduce the cycle time and increase the productivity of hot-press forming process.

**APPENDIX F**  
**JOINT CONFERENCE IMEC & APCOMS 2015**

Experimental validation for hot stamping process by using taguchi method

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**ABSTRACT**

Due to the demand for reduction in gas emissions, energy saving and producing safer vehicles has driven the development of Ultra High Strength Steel (UHSS) material. To strengthen UHSS material such as boron steel, it needed to undergo a process of hot stamping for heating at certain temperature and time. In this paper, Taguchi method is applied to determine the appropriate parameter of thickness, heating temperature and heating time to achieve optimum strength of boron steel. The experiment is conducted by using flat square shape of hot stamping tool with tensile dog bone as a blank product. Then, the value of tensile strength and hardness is measured as response. The results showed that the lower thickness, higher heating temperature and heating time give the higher strength and hardness for the final product. In conclusion, boron steel blank are able to achieve up to 1200 MPa tensile strength and 650 HV of hardness.

**APPENDIX G**  
**INTERNATIONAL CONFERENCE ON NANOSCIENCE AND**  
**NANOTECHNOLOGY 2018**

Numerical simulation of hot press forming process for quenchable sheet metal between nanocoolant and chilled water

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**ABSTRACT**

This paper presents the numerical simulation of hot press forming process by introducing nanocoolant compared with chilled water into the cooling channel system of HPF tool. By using thermal analysis, the heat distribution between heated blank and tools are evaluated. The thermal analysis results show that the average enhancement of nanocoolant cooling rate is measured to be 30.2% higher compared with chilled water. The simulation data are validated with experimental hot press forming results. This study recommends nanocoolant for application in cooling channel system of hot press forming process.

**UMP**

## LIST OF PUBLICATION

### ISI Indexed Journals:

**Lim, S. K.,** Azmi, W. H. & Yusoff, A. R. (2016). Investigation of thermal conductivity and viscosity of Al<sub>2</sub>O<sub>3</sub>/water–ethylene glycol mixture nanocoolant for cooling channel of hot-press forming die application. *International Communications in Heat and Mass Transfer*, 78, 182–189. **Published ISI Q1 (IF 3.718)**

### Scopus Journals:

Zamri, M. H., **Lim, S. K.** & Yusoff, A. R. (2016). Experimental validation for hot stamping process by using taguchi method. *IOP Conf. Series: Materials Science and Engineering*, 114, 012033. **Published**

**Lim, S. K.,** Zamri, M. F. & Yusoff, A. R. (2017). Numerical simulation of hot press forming process for quenched boron steel between nanocoolant and chilled water. *International Conferences on Nanoscience and Nanotechnology 2018*. **Accepted**

### Award:

**Gold Medal Award** (Manufacturing Engineering) in Creation, Innovation, Technology Research Exposition, CITREX 2017, Gambang, Pahang, Malaysia.

Yusoff, A. R. & **Lim, S. K.** (2017). Nanocoolant for improving cooling channel performance in hot press forming die.

**Silver Medal Award** (Manufacturing Engineering) in International Invention, Innovation and Technology Exhibition, ITEX 2017, Kuala Lumpur, Malaysia

Yusoff, A. R. & **Lim, S. K.** (2017). Nanocoolant for martensitic material transformation.