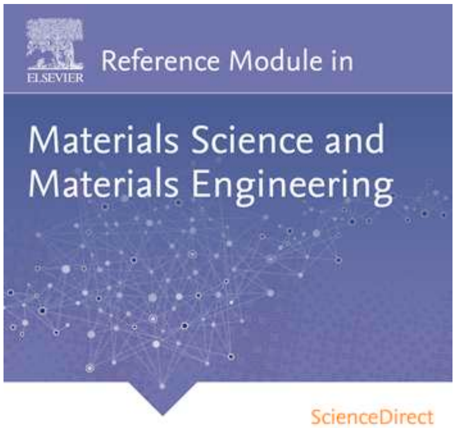

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Microstructure and Mechanical Properties of Boron Sheet Metal Steels in Hot Press Forming Process With Nanofluid as Coolant

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1 Introduction

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 [1]. Most of the UHSS materials are having high mechanical properties as shown in **Fig. 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 [2]. Hence, to overcome these problems of high strength steel forming, researchers and industries are actively developing hot press forming (HPF) technologies [3]. 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 [4,5]. 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 [2]. 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 as depicted in **Fig. 2**.

Naderi reported that boron steel, 22MnB5 produced a fully martensitic microstructure after using HPF process [6]. **Fig. 3** shows the boron steel has a tensile strength of approximately 600 MPa at the initial state. Higher ultimate tensile strength can be attained

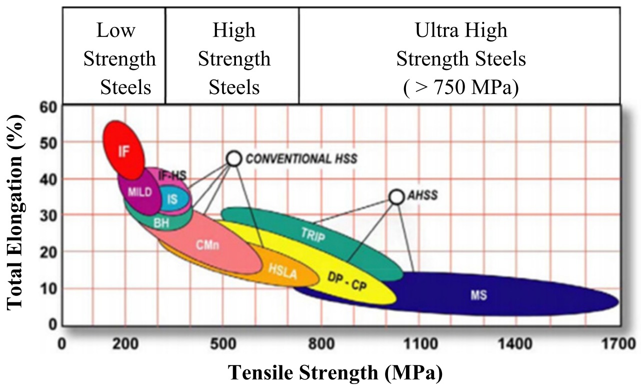

Fig. 1 Tensile strength between UHSS and typical sheet metal. Reproduced from Turetta, A., 2008. Investigation of Thermal, Mechanical and Microstructural Properties of Quenchenable High Strength Steels in Hot Stamping Operations (PhD Thesis). Italy: University of Padova.

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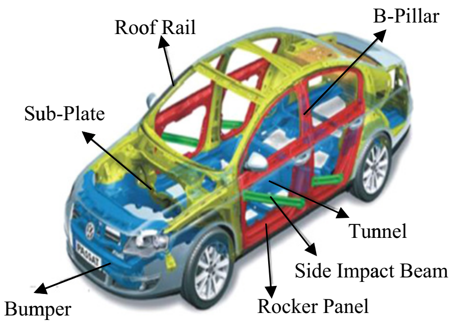


Fig. 2 Hot formed parts in typical middle class vehicle. Reproduced from Karbasian, H., Tekkaya, A.E., 2010. A review on hot stamping. Journal of Materials Processing Technology 210 (15), 2103–2118.

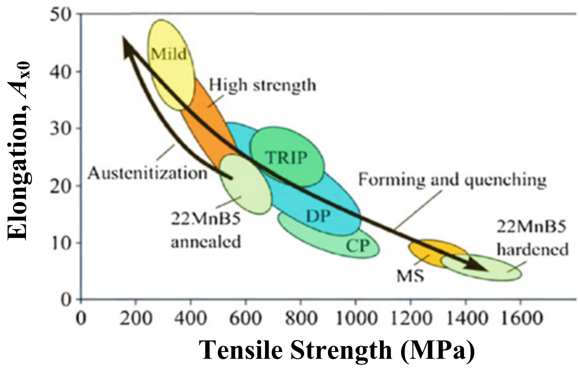


Fig. 3 Mechanical properties of boron steel before and after hot press forming process. Reproduced from Karbasian, H., Tekkaya, A.E., 2010. A review on hot stamping. Journal of Materials Processing Technology 210 (15), 2103–2118.

by a rapid cooling of the hot forming tool at the cooling rate of at least 27 K s^{-1} [7]. 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 [1].

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 [8]. In the process, UHSS blank is cut into the rough dimension, and the blank is heated up to the required temperature of 900°C for 5 min 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 [1]. 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.

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 K 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, they 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 [9,10]. Thus, the development of nanocoolant technology has expanded considerably over the past several years after the pioneer research made by Choi [10] 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*. 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% [11]. Bayat and Nikseresht 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 [12]. 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 [13].

Introduction of nanocoolants as cooling fluid in the 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. In this paper, mechanical properties of boron steel product in terms of tensile strength and hardness with the microstructure transformation were analysed and compared the between the chilled water and nanocoolants.

2 Boron Steels Sheet Metal Forming

Boron steel is one of the low carbon martensitic steel. A group of researchers stated that after the austenitization process, boron steel sheet metal only could achieve Ultra-High Strength Steel (UHSS) grade [14]. 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 [6]. 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 [15].

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 1**. 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 [16].

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 900–950°C [1]. 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 **Fig. 4**. 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 K s^{-1} [17].

The graph shows that the austenitic phase is continuously transformed into martensitic phase, which starts at the temperature of approximately 400°C during quenching operation and it is fully martensitic transformation at a temperature range of 200–250°C. A group of researchers 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 [18]. 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** concludes the summary of boron steel with different microstructure phases in temperature range from 20 to 1000°C during processing.

Table 1 Chemical compositions of boron steel weight percentage and mechanical properties before and after quenching operation

Alloy element								
	Mn	Si	C	Ti	P	Al	S	B
22MnB5	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

Note: López-Chipres, E., Mejíaa, I., Maldonado, C., Bedolla-Jacuinde, A., Cabrera, J.M., 2007. Hot ductility behaviour of boron microalloyed steels. Materials Science and Engineering A 460–461, 464–470.

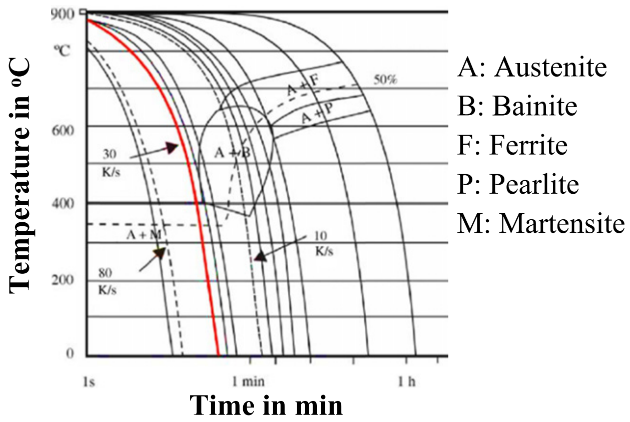


Fig. 4 Temperature, time and transformation diagram of boron steel at various cooling rates. Reproduced from Abdul-Hay, B., Bourouga, B., Dessain, C., 2010. Thermal contact resistance estimation at the blank/tool interface: Experimental approach to simulate the blank cooling during the hot stamping process. International Journal of Material Forming 3 (3), 147–163.

Table 2 Thermal-physical properties of boron steel

Temperature, T (°C)	20	100	200	300	400	500	600	700	800	900	1000
k (W m^{-1} K^{-1})	30.7	31.	30	27.5	21.7		23.6		25.6		27.6
C_p (J kg^{-1} 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

Note: Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

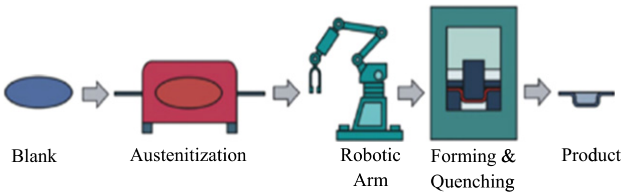


Fig. 5 Direct hot press forming process. Reproduced from Hu, P., Ma, N., Liu, L., Zhu, Y., 2013. Theories, Methods and Numerical Technology of Sheet Metal Cold and Hot Forming, first ed. New York: Springer-Verlag, London.

3 Hot Press Forming

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°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. Finally, the heated blank is formed and quenched in a forming tool enclosure with coolant to produce the fully martensitic transformation part [19]. **Fig. 5** shows the direct HPF process using a robotic arm to transfer the heated blank from the furnace to forming die.

Besides that, **Fig. 6** 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. 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 [19]. In 2009, the researchers stated that indirect HPF focused on quenching and calibration in the press after austenitization operation for complete cold pre-forming part [20]. 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 [21]. Product designs of indirect HPF begin with cold pre-forming dies design before heating up the blank in the furnace and HPF process.

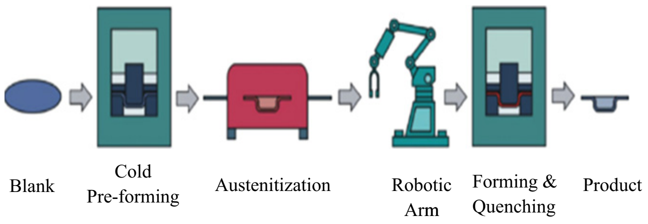


Fig. 6 Indirect hot press forming process. Reproduced from Hu, P., Ma, N., Liu, L., Zhu, Y., 2013. Theories, Methods and Numerical Technology of Sheet Metal Cold and Hot Forming, first ed. New York: Springer-Verlag, London.

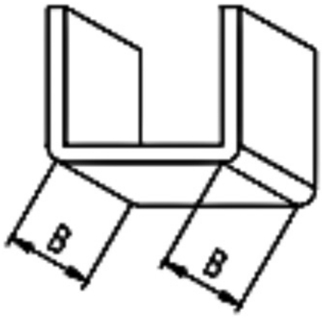


Fig. 7 Hat-shaped profile bending operation. Reproduce from Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

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 [22]. 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 [23]. **Fig. 7** 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 [24]. In this project, hat-shaped profile bending as a sheet metal blank in HPF process for introducing nanocoolants of improvement cooling channel performance.

Hat-shaped sample with tensile shape specification was used because the instrumentations in laboratory limited to hot 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 for the experiment with the blank size dimension of 280 mm × 100 mm × 1.8 mm [25]. **Fig. 8** presents the process flow for hat-shaped samples fabrication by using cold dies as pre-forming operation with a mechanical press machine model OCP 80.

3.1 Nanocoolants as Cooling Fluid for Hot Press Forming

Methods of nanocoolant preparation by using aluminium oxide (Al_2O_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 [26]. The nanocoolant for hot press forming (HPF) experiment was prepared in bulk quantities with volume of 20 l. **Fig. 9** shows the process flow of preparation of Al_2O_3 nanocoolants for investigation of heat transfer distribution in HPF process. After the specific concentration of nanoparticles was calculated by Eq. (1), the Al_2O_3 nanoparticles were weighted by using weighing machine in the material laboratory before being dispersed in the base fluid.

$$\varphi = \frac{m_p/\rho_p}{m_p/\rho_p + m_{bf}/\rho_{bf}} \times 100 \tag{1}$$

Nanocoolant was prepared by synthesizing nanoparticles with base fluid by using mechanical stirrer. The dispersion of pre-calculated weight of Al_2O_3 nanoparticles in water-ethylene glycol mixture was extended by immersing it in an ultrasonic homogenizer for one hour due to the sonication time period influenced the thermal physical properties of nanocoolants [27]. 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 l was completely homogenized and well mixed

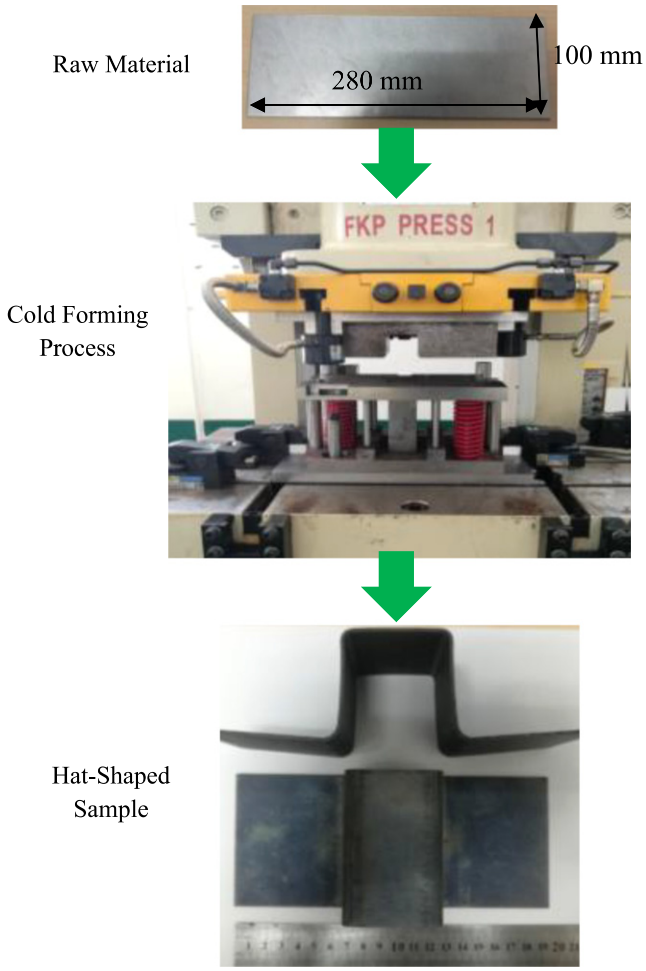


Fig. 8 Hat-shaped samples fabrication from cold forming. Reproduced from Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

with a mechanical stirrer. As review from a group of researchers stated that nanocoolant was uniformly dispersed and well mixed was compulsory for effective production or reproduction of enhanced properties and interpretation of experimental data [28].

In this research experiment, the nanocoolant was synthesized in volume concentration of 0.8% Al_2O_3 nanoparticles were dispersed in the water-ethylene glycol based mixture with 60%:40% volume ratio of 20 l 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 because of introducing surfactants might affect the thermal physical properties of nanocoolants [29]. Serebryakova *et al.* and Zakaria *et al.* also synthesized their nanocoolants without adding surfactants and they found that nanocoolant were in a stable condition [30,31].

3.2 Hot Press Forming Testing

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 [19]. Several researchers Hoffmann *et al.*; Karbasian *et al.* and Naganathan *et al.* had elaborated on the HPF process. The sheet metal was heated up to approximately 900°C for 5 min heating time and then placed in an enclosure cooled die [1,21,32]. The cooling channel has a convection heat transfer coefficient of 7813 W m^{-2} K^{-1} of nanocoolant with a bulk temperature of 25°C. Besides that, chilled water with 4700 W m^{-2} K^{-1} of convection heat transfer coefficient also used to cool down the dies and to allow the quenching operation to transpire.

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. Some researchers conducted 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 [33,34]. Furthermore, the heat transfer distribution experiment was performed to evaluate the cooling rate of the HPF dies.

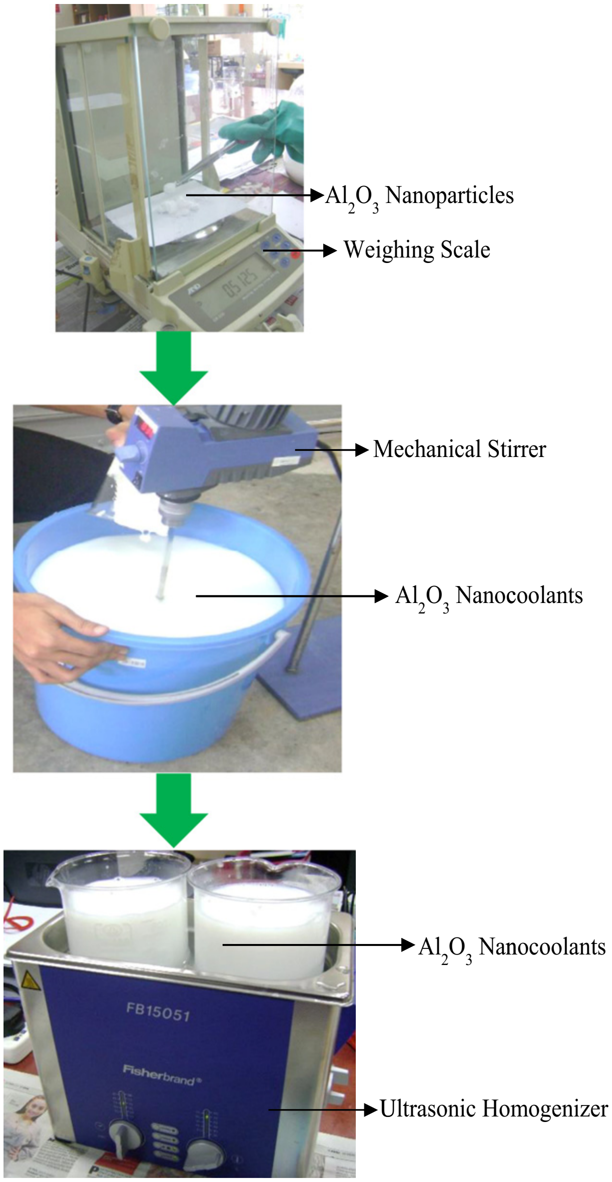


Fig. 9 Al_2O_3 nanocoolant preparation by using ultrasonic bath heater. Modified from Lim, S.K., Azmi, W.H., Yusoff, A.R., 2016. Investigation of thermal conductivity and viscosity of Al_2O_3/water-ethylene gylcol mixture nanocoolant for cooling channel of hot press forming die application. International Communications in Heat and Mass Transfer 78, 182–189.

Lim 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 [35]. 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 min time period [1]. Mori *et al.* found that 5 min of heating time was sufficient to attain the optimum martensitic content in the quenched samples with a maximum hardness of approximately 470 HV [36,37]. As for the quenching time during HPF process, the time interval was 3, 5, 8 and 10 s, respectively.

The hat-shaped blank was heated up 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. 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. Finally, the hot formed part will be measured and analysed by conducting the hardness measurement, tensile test and metallographic study of the microstructure. Fig. 10 shows 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

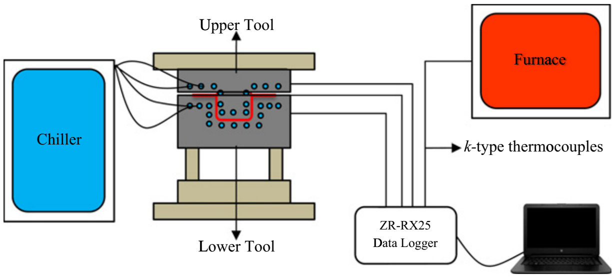


Fig. 10 The experimental equipment setup for hot press forming process. Reproduced from Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia : Universiti Malaysia Pahang.

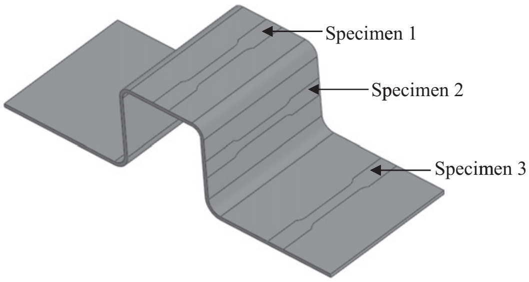


Fig. 11 Location of hot formed samples for tensile specimen, hardness test and microstructure analysis. Reproduce from Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

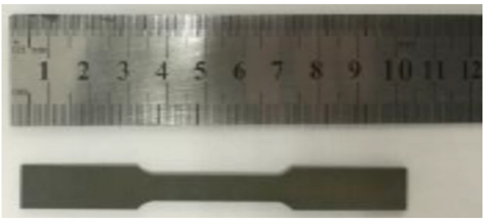


Fig. 12 The dimension of specimen for mechanical testing and microstructure analysis. Reproduce from Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

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.

3.3 Microstructure and Mechanical Properties Measurement

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 Fig. 11. According to the standard of ASTM E 8M, specimens with a total length of 100 mm were cut into 33 mm × 5 mm × 1.8 mm gauge size, which maintained for every tensile test experiment as shown in Fig. 12. Then, the tensile test specimen was clamped by using the upper and lower jaw of the Universal Tensile Machine (UTM) as depicted in Fig. 13. 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 [38].

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 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 min 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 to 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

(a) (b)

Fig. 13 Tensile strength measurement with Universal Tensile Machine. Reproduce from Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

with the hardness readings measured by a test force of 1 N [39–41]. The diamond shapes for the reference sample was produced on the surface and it was measured by using the Vickers Micro-hardness Tester Machine.

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. The inaccuracy of about ±5% in measuring quantitative area fractions of different phases was reported in the software manual [42]. 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 2–5 s time period [41]. The microstructure morphology was observed with a magnification of 20×, 50× and 100× 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.

4 Microstructure and Mechanical Properties Transformation

4.1 Hot Press Forming Microstructure Transformation

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 [43]. 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 3**. At 100× magnification, the as-received sample contained the mixture of pearlite phase and 73%–77% ferrite and a small amount of carbide. Nuraini *et al.* stated that as-received boron steel exhibited pearlite phase located at ferrite grain boundaries [42]. In this phase, ferrite-pearlite microstructure existed and brittle fracture in the sheet metal steel in the state [44]. For hot pressed samples at 100× 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.* stated that brown colour represented martensitic phase after the image was taken to quantify the area fraction [14]. The figure indicated that short holding time was insufficient to enable a martensitic transformation. Löbbe *et al.* 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 K s^{-1} were feasible to reach the martensite start and develop a martensitic microstructure, which was determined and agreed by Naderi *et al.* [41,45].

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.* and Löbbe *et al.* who showed that higher

Table 3 Micrographs of boron steel blank with several quenching time periods

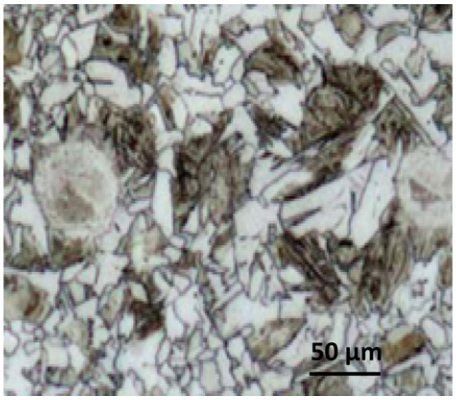
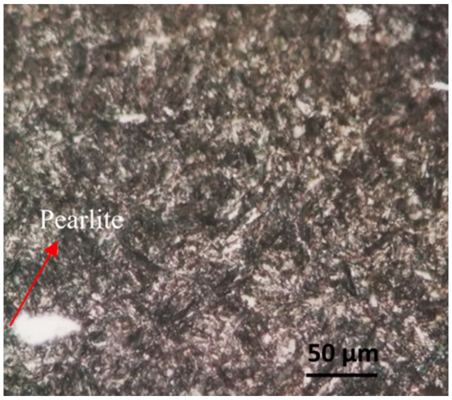
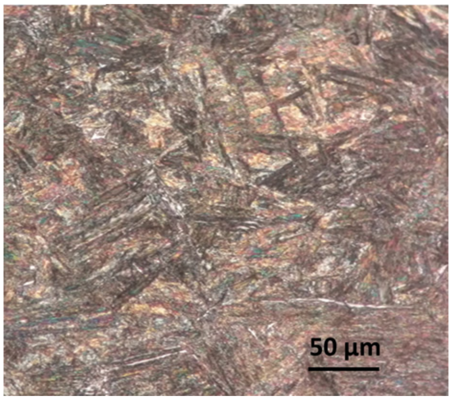
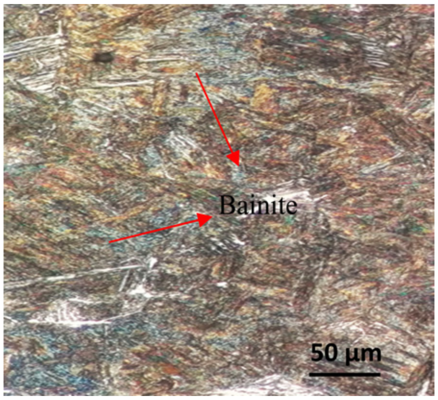
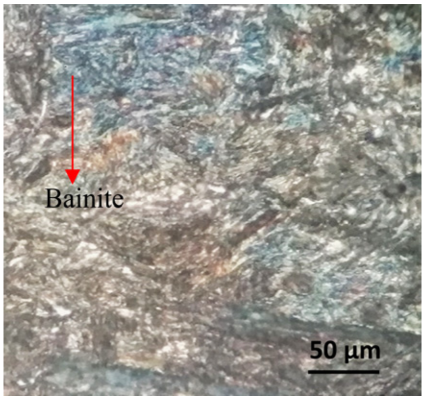
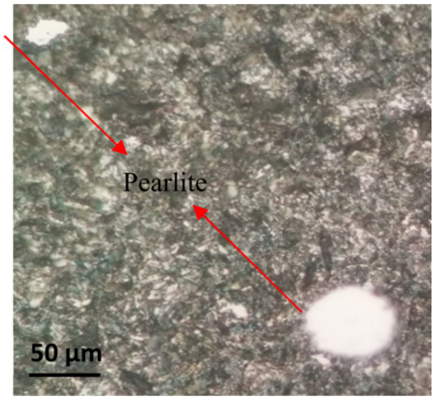
Cooling fluid	*Microstructure*	*Evaluation*
As-received		Material: Boron steel Microstructure: Ferrite and Pearlite
Nanocoolant		Material: Boron steel Temperature: 900°C Holding Time: 3 s Microstructure: Pearlite and Martensite
Nanocoolant		Material: Boron steel Temperature: 900°C Holding Time: 5 s Microstructure: Martensite
Nanocoolant		Material: Boron steel Temperature: 900°C Holding Time: 8 s Microstructure: Martensite and Bainite

Table 3 Continued

Cooling fluid	*Microstructure*	*Evaluation*
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

Note: Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

cooling rate and sufficient or long holding times cause a reduction of the martensite start temperature [41,46]. 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 [45]. 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 [41].

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 after 8 s of quenching time [14]. 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. Löbbe *et al*. stated that the higher temperature and longer holding time caused a grain growth of hot formed sample [41]. 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 [18]. 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*. 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 [41]. Hence, the critical cooling rate was the major controlling factor of martensite formation but rather increasing of short holding time and temperature.

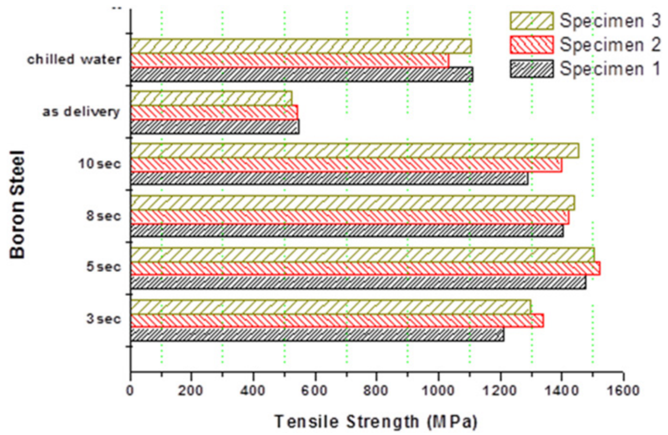


Fig. 14 Ultimate tensile strength of blank samples at several cooling conditions. Reproduce from Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

4.2 Hot Press Forming Mechanical Properties Transformation

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 **Fig. 14**. 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. Researchers 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 [39]. However, the tensile strength value of the boron steel was decreased gradually when increased the quenching time. According to Löbbe *et al.* 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 [41]. This phenomenon could be explained by the producing lath-shaped structure because a finer structure and grain boundaries impede dislocation motions.

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 [41]. 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.* the value of tensile strength after quenching process was around 1400 MPa due to the specimen showed an increase of the martensitic microstructure [20]. Srithananan *et al.* 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 [14]. The tensile strength of boron steel as received stage was low due to the bigger portions of ferrite phase microstructure were detected [6]. However, the ultimate tensile strength value variations of the investigated hot formed specimens had been anticipated. Naderi *et al.* presented that a decreased in tensile strength value, because of the martensite volume fraction reduction [45]. The martensitic phase microstructure due to its high hardness was brittle and low ductility. Moreover, Wang *et al.* 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 [47]. 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.

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 **Fig. 15**. The hardness values were measured by using Vickers method according to DIN EN ISO 6507-1. 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.

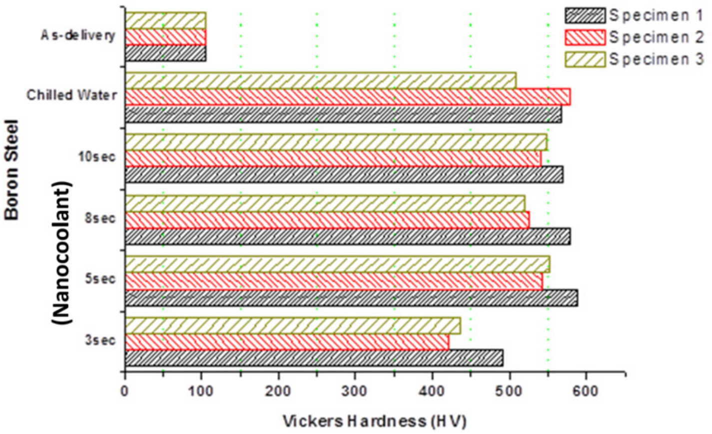


Fig. 15 Hardness value of hot pressed samples at several cooling conditions. Reproduce from Lim, S.K., 2018. Investigation of Nanocoolant Based Al_2O_3 for Improving Cooling Performance in Hot Press Forming (MSc Thesis). Malaysia: Universiti Malaysia Pahang.

Hardness properties of the hot pressed boron steels as different quenching mediums and quenching duration were responding to micrograph as shown in **Table 3**. The highest Vickers hardness value of 588 HV 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 HV. 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 [48]. The martensitic structure would lead to an elevated strength of the boron steel due to an increase in dislocation impedances [39,47].

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.* 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 [49]. 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. Merklein *et al.* found that the hardness value of hot pressed part was around 514 HV [20]. Naderi *et al.* obtained the highest hardness value of approximately 600 HV in their sample of steel-A with different carbon contents [45]. Namklang *et al.* produced the hot pressed part with Vickers hardness value of 550 HV in the bottom area region [48]. Since, the hardness value obtained was higher compared to the normal trend, the results were acceptable.

In this study, thermal analysis was conducted for the heated blank, upper die and lower die of the HPF tool. This approached 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.* [48]. 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.

5 Conclusions

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 1600 MPa 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.

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