

A Study of Microstructural Evolution of AISI D2 Tool Steel during Direct Partial Remelting Method

Fadzilah Adnan, Zainuddin Sajuri, and Zaidi Omar

Department of Mechanical and Materials Engineering, Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Email: fadzilah83adnan@gmail.com.my, zsajuri@ukm.edu.my, zaidiomar@ukm.edu.my

Abstract—Tool steels are difficult to weld using conventional fusion welding process. Therefore, in this study, a thixotropic property of metal was utilized to join an AISI D2 tool steel by using uncommon direct partial remelting (DPRM) method. The aim of this study is to analyse the effect of DPRM on microstructural evolution and hardness property of the welded joint of D2 tool steel. The microstructural analysis showed that the inherent carbides in D2 tool steel fully dissolved when the heating temperature reached between 1250 °C and 1280 °C. Overall, the grain size and shape factor of DPRM samples were within the limit of semisolid processing parameter which are 100 µm and 2, respectively. Based on the microstructural evolution analysis, grain size, shape factor, and hardness value obtained, this study successfully affirmed that with suitable globular microstructure and high bonding quality a sound weld joint of D2 tool steel components can be attained at temperature range of 1280 °C, 1300 °C and 1320 °C.

Index Terms—DPRM, microstructure, hardness, liquid fraction, D2 tool steel

I. INTRODUCTION

AISI D2 tool steel is a high-carbon and high-chromium tool steel. The advantages of D2 tool steel are its characteristics which are their strength, high wear resistance and high hardness after hardening process. Conventional joining methods are not easily applicable due to fact that of high carbon content and high mechanical properties of these steels. Thus, the semi-solid metal (SSM) processing is presented as an auxiliary. The thixotropic property [1] was identified by Spencer et al. (1972) during his study. SSM is having attention around the world due to its advantages in ability to facilitate a high-quality production at a relatively lower cost compared to the conventional process. For example, fewer defects related to solidification processes in a casting process can be improved in the SSM processing. While for forging process, the required forming loads are much lower for the SSM processing, and thus complex shaped parts can be produced net or near-net in a one-step process [2, 3]. Direct Partial Remelting is a method of joining two metals in thixotropic property (Mohammed, M.N., 2013). This process is able to

produce homogeneous properties with a high-quality surface and avoids the creation of a dendritic microstructure in the join zone. In other words, a microstructure containing globule solid particle in a liquid matrix has been considered to be the most appropriate microstructure for the semi-solid forming due to its high fluidity [4]. If enough wetting can be achieved along the grain boundaries, the initial shear during the forming process may cause the grains to flow pass each other, and the semi-solid slurry to flow thixo-tropically.

II. EXPERIMENTAL PROCEDURE

A. Materials

The material used in this work is AISI D2 cold-work tool steel. The material was supplied by ASSAB Sdn Bhd after soft annealing process which had been heated to 850 °C, followed by cooling at 10 °C/h to 650 °C and finally continued with air cooling. The chemical composition of the starting material was acquired by Arch Spark Spectrometer technique is shown in Table 1 which is within the required standards [5].

TABLE I. CHEMICAL COMPOSITION OF AISI D2 (WT. %) COLD WORK TOOL STEEL

	C	Si	Mn	Cr	Mo	V	Fe
Test	1.6	0.254	0.254	10.8	0.826	0.826	Bal

JMatPro (Java-based Material Properties) is simulation software that been developed by Sente Software Ltd. This software aims to build up wide range of materials properties of alloys by integrating theoretical materials properties models and database of software structure with a quantitative calculation for the requisite materials [6]. In this particular work, this software is used to estimate the liquid fraction as well as the solid and liquid temperature of the investigated starting materials. Application of an equilibrium solidification thermodynamic modelling route that has led to the ability to predict a number of critical and physical properties for various alloys is a base for the software calculation. DTA measurement was conducted to estimate liquid fraction as well as solidus and liquidus temperatures of the materials. TG/DTA Perkin Elmer was used to analyse the heating of

D2 tool steel with heating rate 10 °C/min. The sample was cut into small pieces with total weight of 50–100 mg with alumina as reference material.

B. Direct Partial Remelting

As received D2 starting materials were isothermal reheat with a protective atmosphere (argon gas) in the Vistec tube furnace with Carbolite Gero 301 PID controller with single ramp to set point and process time. The temperature setting of sample as per shown in Fig. 1.

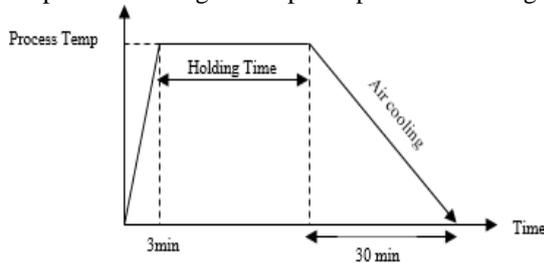


Figure 1. Temperature setting of sample in the Vistec tube furnace

The materials were cut into a sample size of 10 mm x 10mm x 20 mm. The purpose for this isothermal reheating experiment been conducted is to determine the appropriate heating conditions for a successful thixojoining. Besides, the microstructural evolution response to the semi solid state temperature will also been discovered including the grain size and shape factor of the microstructural using the Image J software. In addition, scanning Vickers hardness map was conducted on the sample interface.

The temperatures had been set up for these studies were 1250 °C, 1280 °C and 1300 °C and 1320 °C respectively and held for 0 to 15 minutes (with interval 5 min). The isothermal re-heating experiments were performed using a vertical, high-temperature carbolite furnace, which capable to reach a maximum temperature of 1600 °C. The samples were placed on a ceramic plate of 5mm thick, which had been placed in the middle of the furnace. Both of the top and the bottom of the furnace were sealed to minimize the heat loss. A chromed wire was used to pull out the sample form the furnace to the air cooling to room temperature after the required temperature and the holding time. The selected temperatures were based on the DTA test (10%-40% of liquid fraction value).

C. Microstructural Evolution and Image Analysis

The samples of direct partial re-melting were grinded and polished to acquire a clean and mirror surfaces. Afterwards, the samples were etched by using Villela reagent (1 g picric acid, 5 mL hydrochloric acid and 95 mL ethyl alcohol) to expose their microstructures. The microstructural evolution was captured by using the Olympus optical microscope and a Hitachi S3400N scanning electron microscope (SEM), equipped with energy dispersive spectroscopy (EDS). The image analyses were conducted by using the Image J software to determine the shape factor (SF) and average grain size (GS). The average grain size of primary particle was calculated by using the mean lineal intercept method (after the ASTM E112-96 standard) was defined as

$[\sum 2(A_i/\pi)^{1/2}]/N$, where A_i is the area of each particle and N is the total number of particles in each image [7]. The shape factor was defined as $4\pi A/P^2$, where P is the perimeter, A is the area of the particle (the shape factor of a circle is equal to one) [8]. The grains are completely globule when value of F is equal to 1, while it is more complex when F value is greater than 1[9]. According to Hirt et al. (2009)[10], the ideal semisolid average grainsize should not exceed 100 μm and shape factor must not be greater than 2. The effects of temperature change and holding time on microstructure evolution were studied in this work.

III. RESULT

A. Starting Material

The as-received material consists of carbides in a ferrite matrix as per Fig. 2. This structure is well known to be found in as-annealed tool steel [5], hence confirming that soft annealing treatment was carried out as described by the material supplier. C, V and Cr are mainly composition of M_7C_3 carbide while for $M_{23}C_6$ carbide are rich in C, Cr and W. The SEM-EDX analysis had shown the chemical compositions as per shown in Table 2. It shows the increasing amount of element value of Cr, V as well C and decreasing element value Fe significantly indicates the M_7C_3 carbide type. Due to the soft annealing process at 850 °C, there is no appearance of MC carbides due to the dissolution temperature of MC carbide is at 740 °C. According to (Omar et al. 2011a), the amount of $M_{23}C_6$ carbides after cooling to room temperature is negligible as compared to M_7C_3 carbides [11].

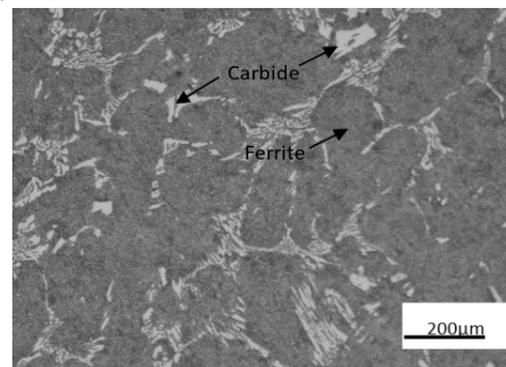


Figure 2. As-received material

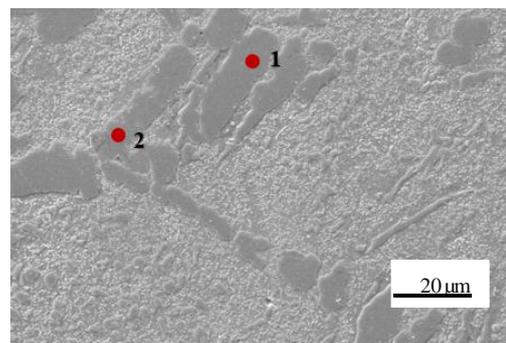


Figure 3. SEM (SE) micrographs of as-received D2 Tool Steel

TABLE II. EDX ANALYSIS RESULT OF THE CARBIDES AND FERRITE MATRIX (WT. %) SHOWN IN FIGURE 3

Point	C	V	Cr	Mn	Fe	Mo	Phase
1	8.34	4.15	42.39	0.35	42.13	1.41	M ₇ C ₃
2	8.62	2.42	17.24	0.35	71.72	0.29	M ₇ C ₃

B. Liquid Fraction Profile

From JMatPro simulation, a phase equilibrium diagram AISI D2 was computed as per illustrated in Fig. 4. The phase transformations of carbides (MC, M₂₃C₆, and M₇C₃), austenite, ferrite and liquid within the temperature range of 0 °C to 1600 °C were showed. It also showed the estimation of solidus and liquidus temperature at almost 1240 °C and 1400 °C respectively. While the austenite phase approximately to begin materializing at 790 °C and melt completely at 1400 °C. The volume of MC carbide is around 1-2% will dissolve completely at temperature 650 °C after reheating while for M₇C₃ carbide completely dissolves at 1251 °C. Based on the Figure, it shows that the M₇C₃ carbides lie in the beginning of semi-solid region. Meanwhile for the M₂₃C₆ carbides, these dissolve completely at around 889 °C. Due to the presence of M₂₃C₆ carbides is relatively small around 5%, therefore the presence of M₂₃C₆ can still be negligible.

Liquid fraction profile (LFP) curves from DTA test for D2 tool steel was showed in Fig. 5. The potential area in terms of temperature sensitivity lies between 1270 °C. and 1340 °C could be the temperature interval for semi solid process. Due to that, these intervals temperature condition has been studied corresponding to the materials microstructures and liquid fractions.

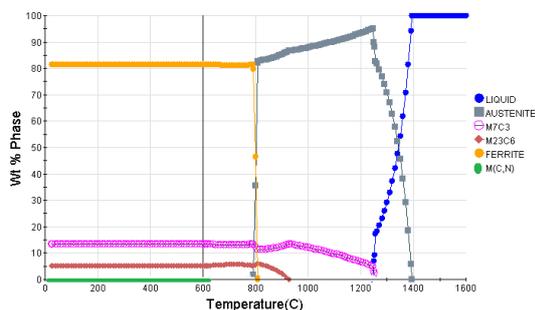


Figure 4. Phase equilibrium diagram of AISI D2 tool steel obtained from JMatPro simulation

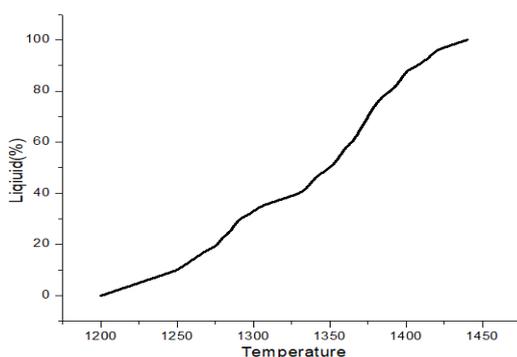


Figure 5. Liquid fraction obtained from DTA

C. Microstructural Evolution

Fig. 6 shows the D2 tool steel at the selected sub solidus temperature was at 1150 °C. In comparison with Fig. 2 and 6, it showed that some of the carbides observed in the earlier have been dissolved. The rest of the undissolved carbides were still contained in bands parallel to the working direction with the fine equiaxed grains also appeared.

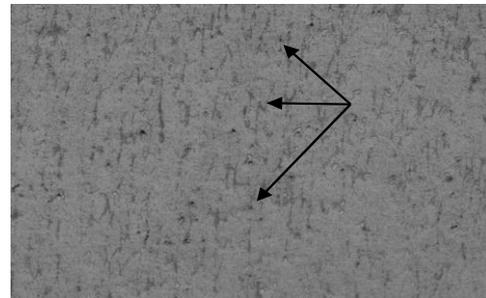


Figure 6. Optical Micrograph D2 after heating to the subsolidus zone

At the temperature of 1250 °C, more original carbide dissolved as compared to those in the sub solidus condition. According to the DTA profile in Figure 5, the liquid fraction is around 10% volume fraction. Through observation, a tremendous amount of non-dissolves carbides presents at this temperature. In addition, comparison can also be made with the JMatPro calculations as per Figure 4, which showed that, the carbide dissolution temperatures obtained from partial remelting experiments were higher. For D2 tool steel, the observed carbide dissolution temperature for M₇C₃ type carbide was found to be in the range of 1250–1280 °C (see Fig. 7a, b).

At a 1280 °C, it shows that the equiaxed grains were transformed into relatively globular grains after the carbide dissolved. It showed that the austenite grains were filled by lamellar eutectic liquid at the grain boundaries. Based on DTA calculation, the liquid fraction average volume fraction was reached by 23%.

When the temperature increased at 1300 °C, a finer globular austenite was exhibited. The transformation into globular grains was supported by the carbide dissolution process. Figure 7c shows the microstructural evolution at temperature of 1300 °C. The eutectic volume fraction is greater than before, which is up to 33% according to liquid fraction of DTA. According to (Omar et al. 2011a), the dissolution of carbides is beneficial, in the sense that it gives a large semisolid interval and low temperature sensitivity [11].

Further heating to the temperature of 1320 °C, the eutectic volume fraction increased up to 38% according to liquid fraction of DTA. Finer globular austenite was exhibited. The increase of volume fraction will also increase the liquid phase which eventually will signify the grain size and globular grain level

Fig. 7 shows the microstructural evolution of isothermal heating of D2 tool steel at temperature 15minutes of 1250, 1280, 1300, 1320 °C. This heating will affect the average grain size and shape factor of the

austenite grain. The average grain size at all of this temperature is found to be in the range of 45-75 μm . It was observed, by increasing the isothermal heating temperature, the grain size will be decreased.

It also can be seen that with the increasing in isothermal temperature, the liquid phase started to diffuse and distribute into the grain boundaries and spread more evenly to create a solid-phase globular structure. According to (Atkinson 2005) spherical shaped grains of this type are one of the most important for the basic requirements for the thixoforming process [12]. Microstructural observation revealed that there was a good arrangement of a eutectic mixture surrounding spherical grains.

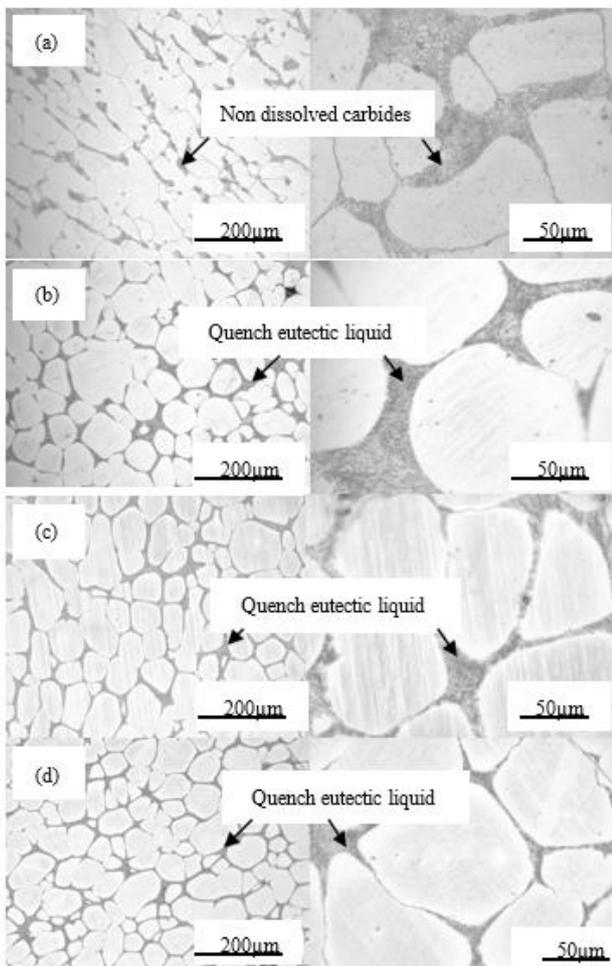


Figure 7. Samples that were treated at (a) 1250 °C, (b) 1280 °C and (c) 1300 °C and (d) 1320 °C holding temperature 5 minutes.

Refer to the Fig. 8, it can be observed that the increasing the temperature, will decrease the grain size. It also reflected to the grain size when increasing the holding time. The grain size value is reflected to the microstructural evolution as per Fig. 7. This finding was supported by M.N Muhammad [13] observed the evolution of relatively spherical particles within a small liquid matrix to the fine globule solid grains in the liquid matrix with the increasing of remelting temperature. Fig. 8 also showed the highest value of grain size is 96 μm at

temperature of 1250 °C and 0 min of holding time. This temperature will not be selected for the thixojoining process due to the value of grain size is almost to the limit. While for the lowest value is 46.26 μm at temperature of 1300 °C with 10 min holding time. Overall value of the grain size showed that they are still in limit of semisolid processing parameter in which below than 100 μm . Fig. 9 shows the shape factor effect of temperature heating and holding time of DPRM. From observation the highest value of shape factor is 0.79 at 1280 °C and 0min of holding time while the lowest value is 0.70 at 1250 °C and 10 min holding time.

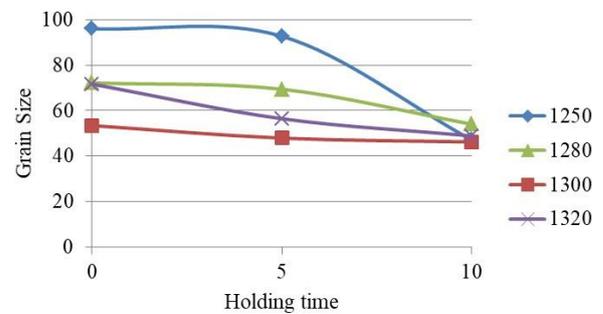


Figure 8. Grain size at various holding time

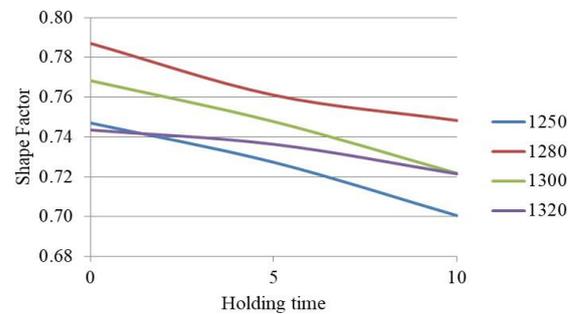


Figure 9. Shape factor at various holding time

In addition, scanning Vickers hardness map was carried out on 20mm x 20mm sections of the direct partial remelting sample interface. The highest hardness value recorded was 400Hv, which had been obtained by isothermal heating at the temperature of 1280 °C for 5 min. Whereas, for the lowest hardness value is 349Hv obtained through isothermal heating at temperature of 1320 °C for 0 min. The hardness value of the received material recorded as 246.6Hv. Details data of hardness effect at various holding time were as per Fig. 10. Effect of the direct partial remelting and holding time will increased of the hardness value.

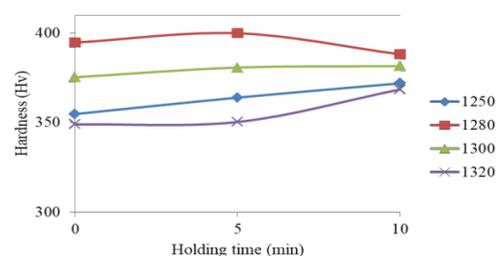


Figure 10. Hardness (Hv) at various holding time

IV. CONCLUSION

The microstructural evolution of received materials of D2 and after direct partial remelting has been analyzed. The received material of D2 tool steels show arrays of carbides distributed in ferrite matrix. At the sub-solidus temperature at 1150 °C, the carbides still clearly in bands parallel to the working direction. From the observation, the carbides are to be fully dissolved at temperature range 1250 °C and 1280 °C. The carbides will be dissolved into the eutectic liquid phase. Penetration will occur if the energy of local grain boundary energy is more than solid liquid energy. Effect of grain size and shape factor to the DRPM had also been observed. In this work, all the samples of DPRM are still in limit of the grain size and shape factor which are 100µm and 2 respectively. The hardness value shows the increased value of DPRM samples compared to the as received materials. The current work confirmed that a joint with suitable globular microstructure and high bonding quality components can be obtained at temperature range of 1280 °C, 1300 °C and 1320 °C after considering the grain size, shape factor and hardness value of the DPRM proses.

ACKNOWLEDGMENT

The authors wish to thank Universiti Kebangsaan Malaysia, Bangi. This work was supported in part by a grant from AP-2015-016.

REFERENCES

- [1] D. B. Spencer, R. Mehrabian, and Flemings, "Rheological behavior of SN-15 pct Pb in the crystallization range," *Metallurgical Transition*, vol. 3, pp. 1925–1932, July 1972.
- [2] Z. Fan, "Semisolid metal processing," *International Materials Reviews*, vol. 47, pp. 49-85, 2002.
- [3] M. N. Mohammed, M. Z. Omar, M. S. Salleh, K. S. Alhawari, and P. Kapranos, "Semisolid metal processing techniques for nondendritic feedstock production," *The Scientific World Journal*, pp. 1-7, 2013.
- [4] J. Li, S. Sugiyama, J. Yanagimoto, Y. Chen, and W. Guan, "Effect of inverse peritectic reaction on microstructural spheroidization in semi-solid state," *Journal of Materials Processing Technology*, vol. 208, pp. 165–170, 2008.
- [5] G. A. Roberts, J. C. Hamaker, and J. R. Johnson, *Tool Steels*, 2nd ed. *American Society for Metals*, Metals Park, 1971.
- [6] N. Saunders, Z. Guo, X. Li, A. P. Miodownik, and J. Ph. Schill é "UsingJmatPro to Model Materials Properties and Behavior," *JOM-The Member Journal of TMS* vol. 12, pp. 60-65, 2003
- [7] M. A. M Arif, M. Z. Omar, N. Muhamad, J. Syarif, P. Kapranos, "Microstructural evolution of solid-solution-treated Zn– 22Al in the semisolid state," *Journal of Materials Science & Technology*, vol. 29, pp. 765–74, 2013.
- [8] F. Czerwinski, "On the generation of thixotropic structures during melting of Mg–9%Al–1%Zn alloy," *Acta Materialia*, vol 50, no 12, pp. 265–81, 2002.
- [9] S. C. Bergsma, M. C. Tolle, M. E. Kassner, X. Li, and E. Evangelista, "Semi-solid thermal transformations of Al-Si alloys and the resulting mechanical properties," *Material Science and Engineering*, vol. 237, 24-34, 1997.
- [10] G. Hirt, L. Khizhnyakova, R. Baadjou, F. Knauf, and R. Kopp, "Semi-solid forming of aluminium and steel introduction and overview," in *Thixoforming-Semi-Solid Metal Processing*, edited by G. Hirt and R. Kopp. USA:Wiley-VCH Verlag GmbH. pp: 1-27, 2009.
- [11] M. Z. Omar, A. Alfian, J. Syarif, and H. V. Atkinson, "Microstructural investigations of XW-42 and M2 tool steels in semi-solid zones via direct partial remelting route," *Journal of Materials Science*, vol. 46, no. 24, pp. 7696-7705, 2011.
- [12] H. V. Atkinson, "Modelling the semisolid processing of metallic alloys," *Materials Science*, vol. 50, pp. 341-412, 2005.
- [13] M. N. Mohammed, M. Z. Omar, J. Syarif, and M. S. Salleh, "Morphological evolution during partial re-melting of AISI D2 cold-work tool steel," *Sains Malaysiana*, vol. 43, no. 8, pp. 1213–1219, 2014.



Fadzilah Adnan, 1983/11/24. She is currently a postgraduate student at Department of Mechanical and Materials Engineering, Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia. She completed her bachelor degree and obtained Master degree in Mechanical Engineering (Hons) in 2010 at Universiti Teknologi Malaysia.



Zainuddin Sajuri, 1971/06/28. He is currently an Associate Professor at Department of Mechanical and Materials Engineering, Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia. He completed his bachelor, master and PhD degree in Materials Engineering at Nagaoka University, Japan. His research interests are reliability of engineering, materials experimental fatigue and fatigue crack growth, fracture mechanics and failure analysis.



Mohd Zaidi Omar, 1971/09/03. He is currently a professor at Department of Mechanical and Materials Engineering, Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia. He completed his bachelor degree at Imperial College. He obtained his master degree at Loughborough University. He obtained his PhD degree in Materials Engineering Sheffield University. His research interests are materials processing, metallurgy, and engineering education.