Optimisation of Compression Moulding Parameter for NR/EPDM Material

R. Izamshah, M. Z. Kasman, M.S. Kasim, M. Rafiq, M.S.A. Aziz

Abstract*: Natural Rubber/Ethylene Propylene Diene Monomer (NR/EPDM) elastomeric has gaining popularity in the automotive industry owing to the fact in term of sustainability. With extensive studies and an increasing number of applications for future advancement, the need for an accurate and reliable guide in processing this type of elastomer has increased enormously. The present work deals with the study of compression moulding parameters (i.e. temperature, pressure, heating time and pressure time) and its effects against NR/EPDM elastomeric mechanical properties (i.e. ultimate tensile strength (UTS), cross-link density and eccentricity error) aim on establishing optimized processing parameters setup. The optimizations are achieved through the Response Surface Methodology (RSM) and mathematical model for each response is developed to access the relationship between the parameters. Adequacy of models is analysed statistically using analysis of variance (ANOVA) in the determination of significant input variables and possible interactions. Lastly, multi objectives optimization is performed through numerical optimization and predicted results are validated. Strong agreement between experimental and the selected solution are found in between 93% and 96%, thus validating the solution as an optimal run condition. The findings suggest that temperature and heating time is the main factor affecting ultimate tensile strength, whereas for cross-link density there is only one significant parameter which is temperature. UTS and cross-link density decrease with the increases of the temperature and heating time due to the degradation (temperature too high for NR/EPDM working temperature). Therefore, it is recommended to start the process below the NR/EPDM degradation point to avoid the scissoring rubber take place and subsequently improving the mechanical properties

Keywords: Precision manufacturing, NR/EPDM, Compression moulding, Optimisation.

I. INTRODUCTION

The development of NR/EPDM blends has steadily increase due to its superior physical properties and excellent resistance to weathering, in particular ozoning. EPDM has better chemical resistance and weathering oxidation [1] while NR has high strength and good dynamic properties [2-3]. In general, rubber-based products can be produced using variety of processes namely extrusion, latex dipping, compression moulding and calendering. For economical reason most of the mass production rubber products are manufactured by compression moulding technique. However, the main disadvantages of this process are the part inconsistency which is directly related with the process parameter that makes this method less sustainable [4]. Two major problems that are associated with compression of elastomer material are distortion and shrinkage. Due to the flexible nature of rubber material and the fact that it is affected by temperature, it is possible for distortion to occur when the rubber part is removed from the mould through the process of stretching it over a core. There are three important factors related to compression moulding process for rubber i.e. temperature, time and pressure [5]. In addition, the selection of the selected parameter value affects the rubber product quality. If the setting temperature is too high, the properties of the rubber will be modified. But if the temperature is too low, the material will result in low liquidation. In addition, same goes like pressure, if the pressure is too low will leads to bad inter-facial adhesion between NR and EPDM. In contrast, high setting pressure will cause a rubber breakage [6]. It shows that the optimum setting condition for both heating time and pressure time are critical for the success of the rubber component. This is due to the different in thermal linear growth between rubber and mould that affect the shrinkage rate during the curing process and result in porosity. Furthermore, product that manufacture by NR/EPDM usually causes an ozone cracking on the surface of the product and directly reduce the shelf life of that product [7-10]. Figure 1 shows an example of distortion and shrinkage on rubber component due to incorrect pressure and temperature.

Thus, the right selection or optimum parameter selection are crucial for the rubber product quality which will be the main work that will be discovered in this paper. The outcome from this work serve as a basis for advancing the technology related to manufacturing of rubber-based product.

Revised Manuscript Received on May 21, 2020.

***** Correspondence Author

R. Izamshah*, Advanced Manufacturing Center, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 D. Tunggal, Melaka, Malaysia. Email: izamshah@utem.edu.my

M. Z. Kasman, Royal Malaysian Maritime Police, 98700 Limbang, Sarawak, Malaysia Email: muhammadzakikasman@gmail.com

M.S. Kasim, Advanced Manufacturing Center, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 D. Tunggal, Melaka, Malaysia. Email: shahir@utem.edu.my

M.S.A Aziz, Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 D. Tunggal, Melaka, Malaysia. Email: sanusi@utem.edu.my

M. Rafiq, Advanced Manufacturing Center, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 D. Tunggal, Melaka, Malaysia. Email: shahir@utem.edu.my

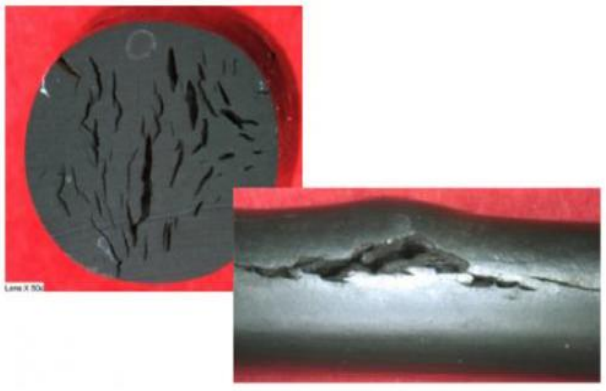

Figure 1: Distortion and shrinkage on rubber component due to incorrect pressure and temperature

II. EXPERIMENTAL DETAILS

A. NR/EPDM Material Composition Tool

The material used in the experiment were categorized in two namely natural rubbers (NR SMR 20 grade) and ethylene propylene diene monomer (EPDM Buna® EPT 9650). Both materials were prepared and processed by Rubber Research Institute of Malaysia (RRIM) according to ASTM D3192 compounding process. Details of the compounding process steps, and the formulation of the NR/EPDM mixture composition were shown in Figure 2 and Table I, respectively.

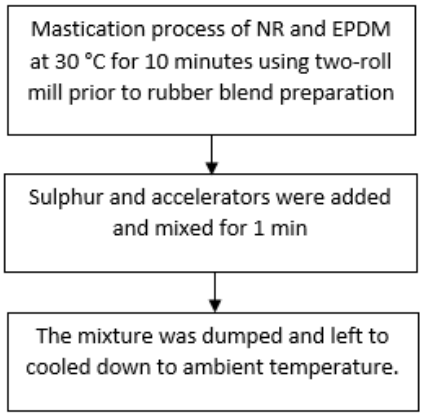

Figure 2: NR/EPDM sample preparation process.

Table- I: NR/EPDM mixture composition

Ingredients	70NR/ 30EPDM/ 10ENR (phr)*
NR SMR 20	70
EPDM	30
ENR-50	10
Zinc oxide	5.0
Stearic acid	2.0
Sulphur	1.5
MBTS[a]	1.0
TMTD[b]	0.3
6PPD[c]	2.0

* part per hundred rubber
[a] 2,20-dithiobis (benzothiazole)
[b] Tetramethylthiuram disulphide
[c] N-(1,3-Dimethylbutyl)-N'-phenyl-p phenylenediamine

B. Compression Moulding Parameter

The compression moulding parameter namely mould temperature, clamping pressure, heating time and pressure time were selected in order to investigate the effects towards NR/EPDM performances. Table 1 tabulated in details the parameters and the level value used in the experimental work.

Table- II: Parameters and levels

Parameter	Levels	
	Lowest	Highest
Pressure (MPa)	10	14.7
Temperature (°C)	140	180
Pressure Time (min)	4	5
Heating Time (min)	4	12

Statistical Response Surface Methodology using Box-Behnken design matrix was used as the design of experiment with totals of 29 runs as depicted in Table III. Compression moulding process were then performed according to the generated runs and were repeated three times. A dedicated mould was fabricated to moulding the NR/EPDM mixture for the subsequent measurement process.

Table- III: Experimental runs

Run	Parameter			
	Mould Temp. (^oC)	Clamp Press. (MPa)	Heating Time (min)	Pressure Time (min)
1	160	12.35	12	5
2	140	12.35	8	5
3	160	14.7	4	4.5
4	160	12.35	8	4.5
5	160	14.7	8	5
6	160	12.35	4	5
7	160	12.35	8	4.5
8	140	12.35	4	4.5
9	160	14.7	12	4.5
10	160	12.35	8	4.5
11	180	12.35	4	4.5
12	160	10	12	4.5
13	160	14.7	8	4
14	180	12.35	8	5
15	160	10	8	4
16	180	10	8	4.5
17	180	12.35	8	4
18	160	12.35	8	4.5
19	140	12.35	8	4
20	140	14.7	8	4.5
21	160	12.35	4	4
22	180	14.7	8	4.5
23	140	12.35	12	4.5
24	160	12.35	12	4
25	160	12.35	8	4.5
26	160	10	8	5
27	160	10	4	4.5
28	180	12.35	12	4.5
29	140	10	8	4.5

C. Performances Measurement

To evaluate the NR/EPDM performances towards the compression moulding parameter, three measurements were made namely ultimate tensile strength, cross-link density and eccentricity.

Maximum force obtained for each sample from the ultimate tensile strength were recorded and tabulated in the analysis table. The samples were prepared into 64 mm x 10 mm dog bone sample size prior for tensile properties measurement according to *ASTM D1822* using Universal Testing Machine (*Toyoseiki Strograph*). The testing was repeated three times to ensure a high confidence level.

For the cross-link density measurement, the NR/EPDM sample was cut into a specific dimension (5x10mm) with the sample thickness of 2 mm. The sample, then, was immersed in the gasoline liquid at normal ambient temperature for 24 hours. Once finished, the samples were removed, and all the sample surfaces were wiped with a clean tissue paper before being weighed. The specimens then were dried in an oven with the setting ambient temperature of 60°C and the weight was, then, measured again and the percentage of swelling was calculated according to equation (1) below:

$$Swelling\ percentage = (w_1 - w_o / w_o) * 100 \quad (1)$$

where w_1 is the mass of the sample after the swelling process and w_o is the default weight of samples before immersing in gasoline.

Finally, the eccentricity measurement was performed using roundness testing machine (*Mitutoyo RA-1600*). In general, eccentricity is referred to the degree of a measured circle approaches an ideal one. Eccentricity error is one of the key parameters to evaluate the NR/EPDM part accuracy. The maximum eccentricity value i.e. sample part deviation from the reference point was recorded and input in the analysis as shown in Figure 2.

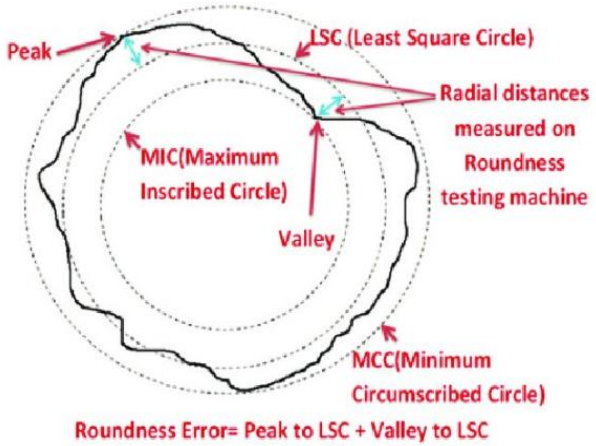

Figure 2: Eccentricity error measurement

III. RESULT AND DISCUSSION

A. Experimental Result

Table IV tabulated the recorded experimental results for ultimate tensile strength, cross-link density and eccentricity based on the 29 runs.

Table- IV: Experimental results

Runs	Ultimate tensile strength (N/m^2)	Cross-link density (1×10^{-7} g/cm^3)	Eccentricity
1	22.81	2.72	4
2	58.75	3.98	2
3	87.87	6.06	2
4	23.75	5.51	3
5	37.19	6.49	6
6	91.25	6.74	1
7	33.44	9.15	1
8	104.06	7.45	3
9	15.62	4.22	3
10	43.75	6.36	1
11	65	5.54	4
12	25.94	5.82	1
13	52.5	6.28	1
14	24.37	4.48	4
15	31.87	6.32	1
16	20.63	4.45	3
17	9.38	3.05	3
18	44.06	6.95	2
19	27.19	6.73	4
20	57.5	6.99	2
21	82.81	6.28	2
22	38.44	7.75	2
23	15.63	5.73	1
24	26.56	6.12	4
25	18.12	5.06	3
26	30.31	5.13	2
27	52.5	6.81	2
28	44.69	4.25	2
29	54.35	4.89	2

B. Effects of Parameter on Ultimate Tensile Strength

The maximum UTS value obtained was 104.06 N/m^2 whereas the minimum was 9.38 N/m^2 with the average of 42.77 N/m^2. The variations of UTS value indicated that the compression moulding parameters affected the UTS response. ANOVA results of the UTS model was found to be significant, with p-value of 0.001. Hence, parameter inputs affected the output. The results were supported by the non-significance of lack of fit where the p-value was 0.3181. Lack of Fit which is not significant shows the chance of no effect of the input to the output is very small. Based on ANOVA (Table- V), the most significant factor that influenced the UTS with the P-Value less than 5% was heating time (C) followed by an interaction between AC term and mould temperature (A). The factors of AD, BD, BC, B, BC, AB, D and CD, however, found to be not significant to the output, these terms, thus, were omitted from the model.

In addition, R^2 value of the model was found to be 0.71 which was considered moderate adequacy fit of the model. The predicted R^2 of 0.46 and the adjusted R^2 of 0.64 were considered a reasonable agreement where the deviation between both values was minimum (less than 0.2).

Table- V: ANOVA result for UTS

Source	Sum of Squares	df	Mean Square	F-Value	P-value
Model	12009	5	2402	11.01	< 0.0001
Mould Temp. (A)	1102	1	1102	5.05	0.0345
Clamping Pressure (B)	450	1	450	2.07	0.1641
Heating Time (C)	9199	1	9199	42.18	< 0.0001
Pressure Time (D)	98	1	98	0.45	0.5084
AC	1160	1	1160	5.32	0.0304
Residual	5016	23	218		
Lack of Fit	4472	19	235	1.73	0.3181
Pure Error	544	4	136		
Cor Total	17025	28			

Based on ANOVA, mould temperature and heating time were the dominating factors affecting the ultimate tensile strength. The UTS reduced as the mould temperature and heating time increase. The reason of this scenario is due to the nature of rubber compound where the elasticity and elongation increased as the temperature increased. Therefore, the tensile decreased. The result varied according to the grade where the tensile of low-grade rubber changed more drastically than high-grade rubber. Figure 3 renders the influence of four factors on the UTS. It shows that heating time (factor C) had the most effect on the responses. Followed by mould temperature (factor A). Both were in the declining trend where the increase of the input decreased the UTS value. However, clamping pressure (factor B) and pressure time (factor D) found to be less effective on the UTS. Both factors were in the positive trend indicating the UTS increased as the parameter value increased.

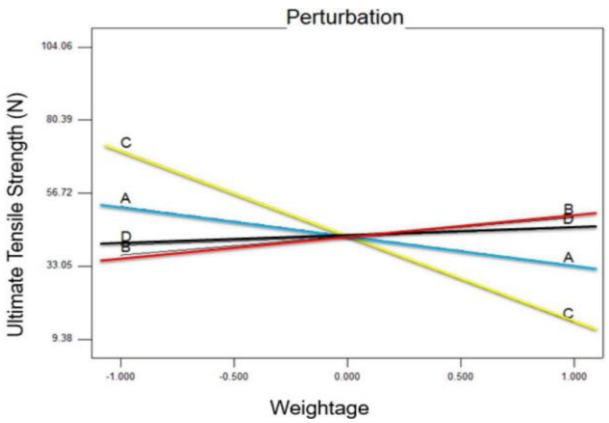

Figure 3: Perturbation plot for UTS

The relationship between ultimate tensile strength value towards the compression moulding parameters can be model in mathematical form and written as:

$$UTS\ (N/m^2) = 389.29 - 2.18A + 2.61B - 40.98C + 5.73D + 0.21AC \quad (2)$$

C. Effects of Parameter on Cross-link Density

The results of rubber cross-link density as indicated in Table IV shows that the minimum cross-link density recorded was 2.72 x 10^{-7} g/cm³ whereas the maximum average value was 9.15 x 10^{-7} g/cm³. Based on ANOVA analysis in Table VI, the cross-link density model was found to be significant with the P-Value of 0.0185 which was less than 5%. it was supported with the lack of fit value which was not significant with the P-value of 0.7375. Among all the parameters, only mould temperature (A) was found to be significant. P-Values less than 0.05 indicated the term significant effects in the design space. The C and D terms could be stated as marginally significant while B term is not significant. The R^2 obtained however, showed less adequacy in predicting response with the value of 0.38.

Table- VI: ANOVA result for cross-link density

Source	Sum of Squares	df	Mean Square	F-Value	P-value
Model	2.81 x 10^{-13}	4	7.024 x 10^{-14}	3.65	0.0185
Mould Temp. (A)	1.69 x 10^{-13}	1	1.69 x 10^{-13}	8.79	0.0067
Clamping Pressure (B)	1.56 x 10^{-15}	1	1.56 x 10^{-15}	0.081	0.7781
Heating Time (C)	5.36 x 10^{-14}	1	5.36 x 10^{-13}	2.78	0.1082
Pressure Time (D)	5.66 x 10^{-14}	1	5.66 x 10^{-14}	2.94	0.0994
Residual	4.62 x 10^{-13}	24	1.93 x 10^{-14}		
Lack of Fit	3.60 x 10^{-13}	20	1.80 x 10^{-14}	0.70	0.7375
Pure Error	1.02 x 10^{-13}	4	2.56 x 10^{-14}		
Cor Total	7.43 x 10^{-13}	28			

Based on the ANOVA results, only mould temperature was considered a significant factor to the cross-link density. Perturbation plot was used to check for the effects of parameters as seen in Figure 4. Factor A (mould temperature) was the steepest among other terms. While factor A, C and D showed a negative trend where the increasing in each factor reduced the cross-link density, factor B (clamping pressure) showed almost constant with no effect on cross-link density by changing this parameter.

The relationship between cross-link density value towards the compression moulding parameters can be model in mathematical form and written as:

$$Crosslink\ density\ (g/cm^3) = 2.21x10^{-6} - 5.93x10^{-9}A + 4.85x10^{-9}B - 1.67x10^{-8}C - 1.37x10^{-7}D \quad (3)$$

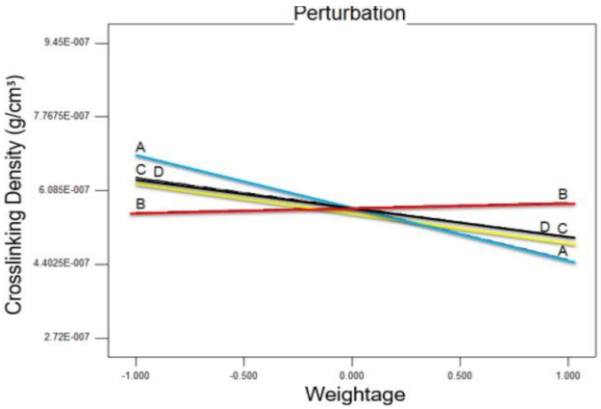

Figure 4: Perturbation plot for cross-link density

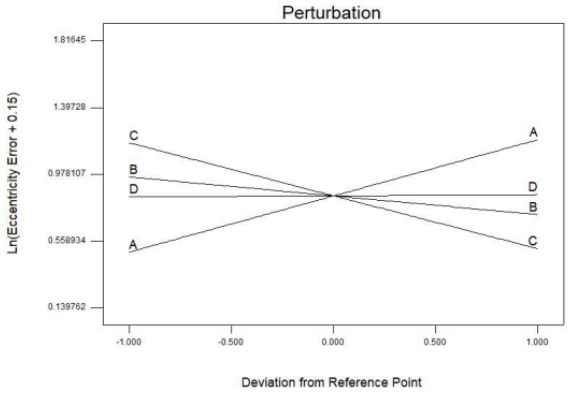

Figure 5: Perturbation plot for eccentricity error

D. Effects of Parameter on Eccentricity

The results of eccentricity as indicated in Table IV shows that the minimum eccentricity error recorded were 1 whereas the maximum error value was 6. Based on ANOVA analysis in Table VII, the eccentricity error model was found to be less significant with the P-Value of 0.0055 and it was supported with the R^2 value of 0.444. lack of fit value which was not significant with the P-value of 0.7375. Among all the parameters, only mould temperature (A) was found to be significant. Perturbation graph in Figure 5 illustrated the relationship of each parameter towards the eccentricity. The increasing of the pressure will increase the eccentricity value. However, for the other parameters are vice versa, the increasing of the temperature, heating time and pressure time will reduce the eccentricity due to the degradation of NR/EPDM itself.

Table- VII: ANOVA result for eccentricity

Source	Sum of Squares	df	Mean Square	F-Value	P-value
Model	3.46	4	0.87	4.79	0.0055
Mould Temp. (A)	1.74	1	1.74	9.61	0.0049
Clamping Pressure (B)	0.20	1	0.20	1.12	0.3011
Heating Time (C)	1.52	1	1.52	8.43	0.0078
Pressure Time (D)	1.15×10^{-3}	1	1.15×10^{-3}	6.39×10^{-3}	0.9369
Residual	4.34	24	0.18		
Lack of Fit	3.42	20	0.17	0.74	0.7117
Pure Error	0.92	4	0.23		
Cor Total	7.8	28			

The relationship between eccentricity error value towards the compression moulding parameters can be model in mathematical form and written as:

$$Ln\ (Eccentricity\ Error) = -0.96982 + 0.01A - 0.05B - 0.08C + 0.01D \quad (4)$$

E. Parameter Optimisation

The optimisation goals are to maximize the desired target for all the response with a higher degree of importance and in this case maximum UTS, maximum crosslink density and minimum eccentricity error. Based on the desire response, the calculated optimisation result was tabulated in Table VIII. The highest desirability index was found to be 0.692 by using 140.2 ^oC mould temperature, 14.7 N/m^2 pressure, 4.5-minute heating time and 4.5 minute pressure time.

Table- VIII: Optimization result

A (^oC)	B (MPa)	C (min)	D (min)	*Desirability*
140.2	14.7	4.5	4.5	0.692

IV. CONCLUSION

This study has investigated the effects of compression moulding parameter on UTS, crosslink density and eccentricity error for NR/EPDM elastomeric. A predictive model for each response associate with the parameter were develop. Finally, optimum parameters as to achieve the best responses was suggested. It shows that, the performance of NR/EPDM product can be control by choosing the right combination of compression moulding parameter. Thus, the right selection or optimum parameter selection are crucial for the rubber product quality. The outcome deliberates from this work serve as a basis for advancing the technology related to manufacturing of rubber-based product.

ACKNOWLEDGMENT

The authors are grateful to Malaysian Ministry of Higher Education and Universiti Teknikal Malaysia Melaka for their technical and financial support under Prototype Research Grant Scheme No: PRGS/1/2019/TK03/UTEM/02/1 and FRGS/1/2018/TK03/02/1.

REFERENCES

1. Botros, S. H., 2002. Preparation and Characteristics of NR/EPDM Rubber Blends. *Polymer Plastics Technology and Engineering* 41(2), pp.341-359.
2. Suma, N. J., R., and Francis, D. J., 1990. Improved Compatibility of Natural Rubber (NR)/Butyl and NR/EPDM Blends by Carboxylating Butyl and EPDM. *Kautschuk und Gummi*, Kunststoffe, 43(12), pp.1095–1097.
3. M. Amran, S. Salmah, A. Faiz, R. Izamshah, M. Hadzley, B. Manshoor, M. Shahir and M. Amri, 2014. Effect of injection moulding machine parameters on the warpage by applying Taguchi method. *Applied Mechanics and Materials*, vol. 699, pp.20-25.
4. Ruksakulpiwat, Y., Sridee, J., Suppakarn, N., & Sutapun, W., 2009. Improvement of Impact Property of Natural Fiber–Polypropylene Composite by using Natural Rubber and EPDM Rubber. *Composites Part B: Engineering*, 40(7), pp.619-622.
5. Zhang H., Datta R.N., Talma A.G and Noordermeer J.W.M., 2008. Blending, Curing and Reinforcement of NR/BR/EPDM Compounds for Tire Sidewall Applications. *Journal of Applied Polymer Science*, University of Twente, pp.2555-2563.
6. Blackley D.C., 1983. Synthetic Rubbers: Their Chemistry and Technology. Applied Science Publishers, pp.208-212.
7. Allen, W. S., Baker, P. N., & Bhatia, S. C., 2009. Handbook of Plastic Technology. Singapore:Alkem Company (S) Pte.
8. M. Amran, S. Laily, H.I.K. Nor, N.I.S. Hussein, M.R. Muhamad, B. Manshoor, M.A. Lajis, R. Izamshah, M. Hadzley, R.S. Taufik, 2014. The effect of EDM die-sinking parameters on material characteristic for aluminium composite using tungsten copper electrode. *Applied Mechanics Materials*, vol. 465, pp.1214-1218.
9. M.A.M Amran, N. Idayu, K.M. Faizal, M. Sanusi, R. Izamshah and M. Shahir, 2016. Part weight verification between simulation and experiment of plastic part in injection moulding process. *IOP Conference Series: Materials Science and Engineering*, vol. 160, no. 1, pp.1-7.
10. Chapman A.V., 2007. Natural Rubber and NR-Based Polymers: Renewable Materials with Unique Properties. Advances in The Field of Elastomers & Thermoplastic Elastomers. *Transport*, 5(8), pp.5-6.

AUTHORS PROFILE

Associate Professor Dr. Raja Izamshah serves more than 15 years both in academic and research at Universiti Teknikal Malaysia Melaka. He received his PhD from RMIT University, Australia. Currently, he is the Head at Advanced Manufacturing Center. His current research interest includes precision machining, numerical simulation and optimization for aerospace and medical fields.

M. Z. Kasman currently work as an inspector at Royal Malaysia Police. He obtained his BEng. degree from Universiti Teknikal Malaysia Melaka majoring in manufacturing engineering. Currently he is pursuing a Master of Science at Faculty of Manufacturing Engineering, UTeM majoring in compression moulding process for natural rubber.

Associate Professor Dr. M.S. Kasim is principle member at the Advanced Manufacturing Center, Universiti Teknikal Malaysia Melaka. He received his BEng from the Coventry University, UK, MSc from the Universiti Putra Malaysia and from the National University of Malaysia. His research interest includes high speed machining on Inconel 718 and process optimization.

Dr. M. S. A. Aziz is currently a senior lecturer at Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka. He obtained his B.Eng (2008), M.Eng (2010) and PhD (2013) from Kanazawa University, Japan. His current research interest includes layered manufacturing, precision and high-speed machining.

M. Rafiq currently work as a researcher at Advanced Manufacturing Center, Universiti Teknikal Malaysia Melaka. He obtained his BEng. degree from Universiti Teknikal Malaysia Melaka and currently pursuing a Master of Science at Faculty of Manufacturing Engineering, UTeM. His research interest is cutting tool technology.