Archives of Materials Science and Engineering

Volume 89
Issue 2
February 2018
Pages 55-63

International Scientific Journal
published monthly by the
World Academy of Materials
and Manufacturing Engineering

Statistical optimization of stress relieving parameters on closed cell aluminium foam using central composite design

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ABSTRACT

Purpose: This study concerns about the influence of stress relieving parameters on the hardness of closed cell aluminium foam using central composite design.

Design/methodology/approach: The responses of three stress relieving parameters: heating temperature, holding time and stabilization temperature are studied and analysed through 20 experimental runs designed according to central composite design. The results of microhardness test corresponded to the microstructural evaluation of closed-cell aluminium foam using optical microscope. Analysis of Variance (ANOVA) technique is employed to study the significance of each parameter on the microhardness property. In this process the design has five levels for each parameter. The stress relieving process of the samples were performed using a vacuum furnace. The hardness test was conducted using a micro hardness tester LM247AT and the microstructure of the samples were obtained using optical microscopy technique.

Findings: It was found that the highest value of hardness of 192.78 HV was obtained when the stress relieving process is set with the following parameters: heating (500°C); holding time (120 min) and stabilization temperature (450°C). Since higher heating temperature and longer holding time produce sample with larger grain size and has an adverse effect on the hardness value.

Research limitations/implications: Liquid metal and powder metallurgical processing still produces a non-uniform and poorly reproducible cellular structure. This cellular structure demonstrates poor quality difference on decomposition and melting temperature, called anisotropic early expansion.

Originality/value: To improve the poor cellular structure quality, stress relieving method is proposed in this study. Stress relieving method can improve the microstructure of the material.

Keywords: Statistical optimization, Stress relieving method, Closed cell aluminium foam, Hardness, Microstructure

Reference to this paper should be given in the following way:

D. Puspitasari, T.L. Ginta, M. Mustapha, N. Sallih, P. Puspitasari, Statistical optimization of stress relieving parameters on closed cell aluminium foam using central composite design, Archives of Materials Science and Engineering 89/2 (2018) 55-63.

METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

1. Introduction

Nowadays, many research in engineering focuses on developing the new material which combines two excellent properties, which is low in density and lightweight. Development of metal foam has enabled us achieving towards this goal [1]. Metal foam is a new material with novel physical, mechanical, thermal, electrical and acoustic properties [2]. The potential applications of metal foams have been applied in automotive industries, lightweight construction materials, silencers, flame arrester, heaters, heat exchangers, catalysts, electrochemical applications, military armour vehicles and aircraft industries [3]. The primary advantages of metal foam are low weight with an excellent combination of mechanical properties such as strength and stiffness. Metal foam absorbs high impact energy, very efficient in sound absorption, electromagnetic shielding, and vibration damping [4]. Energy absorbers were frequently used in vehicles to safeguard passengers and the structure during impact [5].

Liquid metal and powder metallurgical processing are basic methods to produce closed cell aluminium foam [6]. Scientific and industrial interests on powder metallurgical method become significantly increased due to its ability to produce relatively complex shaped foam and sandwich components, and it has the flexibility to choose alloys [7]. This method began with mixing aluminium metal powder and foaming agent [8].

After mixing process, the powder is compacted directly either using a hot pressing, cold working, conform extrusion or powder rolling method. Then, the powder is heated to its solidus temperature to allow decomposition and formation of bubbles. The low-density foam structure of the originally closed cell is gained after cooling [4].

However, this method still produces a non-uniform and poorly reproducible cellular structure [9]. This cellular structure demonstrates poor quality difference on decomposition and melting temperature, called anisotropic early expansion. To improve the poor cellular structure quality, stress relieving method is proposed in this study. Stress relieving method can improve the microstructure of the material. The selection of processing parameters in producing closed cell structure with impaired quality is critical. Therefore, this study focuses on optimizing these parameters to produce improved mechanical properties of closed cell aluminium foam.

2. Methodology

2.1. Materials

Closed cell aluminium foam used in this study included 65.40% Al, 18.70 % Ca, 3.44 % Ti, and 2.29 % Zn. Aluminium foam produced by powder metallurgical method. The method includes mixing the aluminium powder with titanium hydride (TiH_2) and compacted by directly hot or cold processing. Then, heating the precursor greater than its solidus temperature to obtain the originally closed cell aluminium foam [4].

2.2. Central composite design

Design of experiment methodology is widely used to optimize main influence parameters to the response and improvement of the final products [10]. The response surface can be distinctly used to describe the relationship between parameters and response variable [11]. To obtain the optimum parameters there are three steps in Design of Experiment. The first is through statistical design experiments, followed by estimation of coefficients through a mathematical model with response prediction, and completed by analysis of the model's applicability [12]. The central composite design (CCD) was chosen for this study. This design has five levels for each factor [13]. For statistical design experiment, three main factors such as temperature (x_1), holding time (x_2) and stabilization temperature (x_3) were studied using full factorial design, given in Table 1. In the current research, the data analysis for this experiment was conducted using Design of Experiment software program, version 7.1.3, Stat-Ease Inc., Minneapolis MN.

After variance analysis, commonly a second order regression of response variable (y) is achieved in the form of dependent variable (x_{ij}) and the coefficient (β_{ij}) as presented in Equation 1 [14,15].

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} {x_1}^2 + \\ + \beta_{22} {x_2}^2 + \beta_{12} x_1 x_2 + \varepsilon \qquad (1)$$

Analysis of variance (ANOVA) is employed to evaluate the quality of the model fitted by the application. In ANOVA, the evaluation of data set variation is made by studying its dispersion [16]. The test of significant regression is performed in order to determine the linear relationship between the response variable (y) and a regressor variable ($x_1, x_2, \dots, x_k$) [15]. It can be deduced that there is a significant contribution when the *P*-value is adequately small and less than 0.05 (5%) which is commonly used as cut off value [17]. The F_0 is estimated by Equation 2.

$$F_0 = \frac{SS_R/k}{SS_E/(n-k-1)} \tag{2}$$

where F_0 is the test statistic for F-tests, SS_R is regression sum of squares, SS_E is errors sum of squares, k represents as number of factors and n describe as total number of experiment [15].

Table 1.
The CCD with the coded and actual value

Standard Order (Std)	code			Response: Hardness, HV
	x_1	x_2	x_3	
1	500.00	60.00	375.00	161.58
2	600.00	60.00	375.00	141.48
3	500.00	180.00	375.00	117.82
4	600.00	180.00	375.00	139.98
5	500.00	60.00	450.00	192.78
6	600.00	60.00	450.00	144.52
7	500.00	180.00	450.00	183.48
8	600.00	180.00	450.00	125.72
9	465.91	120.00	412.50	144.64
10	634.09	120.00	412.50	119.78
11	550.00	19.09	412.50	164.42
12	550.00	220.91	412.50	129.38
13	550.00	120.00	349.43	104.68
14	550.00	120.00	475.57	153.42
15	550.00	120.00	412.50	114.4
16	550.00	120.00	412.50	105.18
17	550.00	120.00	412.50	122.78
18	550.00	120.00	412.50	107.86
19	550.00	120.00	412.50	118.48
20	550.00	120.00	412.50	114.68

2.3. Hardness measurement and micrograph

The samples were prepared in a vacuum and cut into 10 mm x 10 mm x 20 mm using a slow speed diamond cutter and mechanically ground using 400-1200 grit size silicon carbide paper. The samples were polished with 3 µm diamond paste and etched using Keller's etch (distilled water of 190 ml, HNO_3 of 5 ml, HCl of 3 ml, and HF of 2 ml). These samples were used for microstructure and hardness testing. Leica Application Suite (LAS V4.5) software was used to analyse the micrographs. Microhardness test on cell wall nodes was conducted in according to ASTM E3-11. Five hardness readings were taken on each node and the average was calculated. Microhardness test was conducted using microhardness tester LM247AT with a load 150 mN and loading time of 15 s.

2.4. Compressive testing

The compression test specimens are cut into 30 mm x 30 mm x 50 mm using according to standard test method for compressive properties of rigid cellular plastics (ASTM: D 1621-00). The quasi static compression test was conducted by universal testing machine (max load 5 kN) with computer interface for data acquisition and control. Test was carried out on under displacement control with a cross head speed of 1 mm s^{-1}. The nominal stress was generated by dividing the load with the original cross section area, and the nominal strain was determined by dividing the cross head displacement with original section high of the specimen [18]. The compressive strength (σ_y) determine on the compressive curves as the intersection of the loading and plastic deformation plateau [19].

3. Results and discussion

According to the determined design, 20 experiments were randomly carried out. The results of this study were exposed to variance analysis for the response variable and a regression model was established.

3.1. ANOVA result and regression model

Analysis of variance (ANOVA) is used to study the effects of process parameters on each response in order to determine the significance and suitability of the model which includes the interaction between process variables [14]. Table 2 presents the ANOVA result for surface reduced quadratic model. The response for hardness has F-value of 12.73 and the corresponding p-value of 0.0001 which is less than 0.05. This designates that the model is substantial with only 0.01% chance such that the "Model F-value" could have happened due to noise.

High degree of correlation between the response and the independent variables present by coefficient determination (R-squared and adjusted R-squared) [12]. In this study, the value of R squared is 0.8813 and the adjusted R squared of 0.8120. It means that 88.13% of the variation in the response is explained by the independent variables. The predicted hardness value versus actual hardness value is shown in Figure 1.

Table 2.
ANOVA for response surface reduced quadratic model

Source	Sum of squares	DF	Mean squares	F value	p-value Prob > F
Model	10837.37	7	1548.20	12.73	0.0001
x_1 −	1555.90	1	1555.90	12.79	0.0038
x_2 −	1281.46	1	1281.46	10.53	0.0070
x_3 −	2057.08	1	2057.08	16.91	0.0014
$x_1 \times x_3$	1460.16	1	1460.16	12.00	0.0047
${x_1}^2$	1254.04	1	1254.04	10.31	0.0075
${x_2}^2$	3039.19	1	3039.19	24.98	0.0003
${x_3}^2$	971.64	1	971.64	7.99	0.0153
Residual	1459.78	12	121.65		
Lack of Fit	1246.57	7	178.08	4.18	0.0674
Pure Error	213.21	5	42.64		
Cor Total	12297.15	19			
R-squared	0.8813		Predicted R squared	0.5479	
Adjusted R Squared	0.8120		Adequate Precision	11.211	

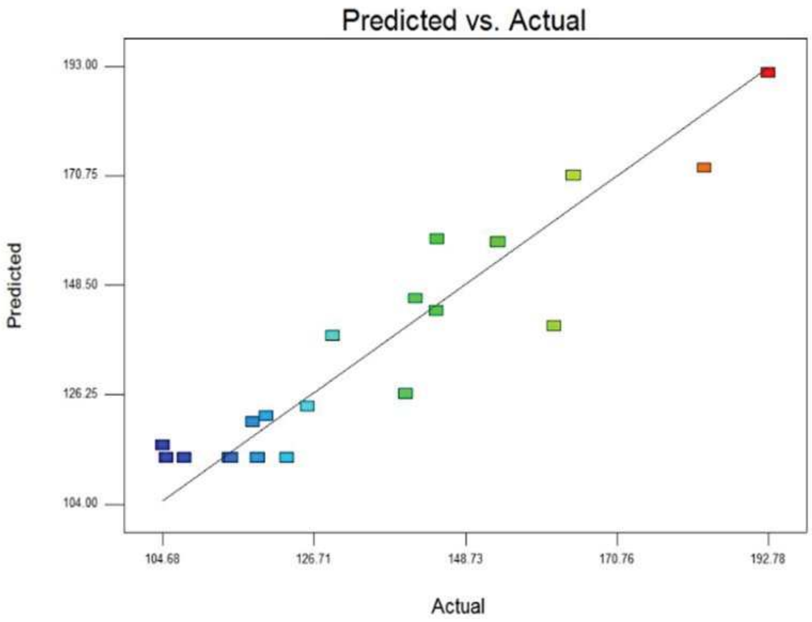

Fig. 1. Relationship between experimental and predicted value

As can be seen in the Table 2, the F-value of Lack of Fit for the model is 4.18. It means that Lack of Fit is not significant relative to the pure error. There is a 6.74% possibility that a Lack of Fit of F-value could occur due to noise. The model will be accepted if the p-value of the F-test for Lack of Fit is not significant (α=0.05) and the model is significant [13]. Figure 3 shows no apparent problem with normality. The function of normal probability plot is to analyse the data normal distribution and is used to assess the assumption of a fixed distribution.

The second order polynomial equation form explains the relationship of the hardness and the stress relief parameters. As a result of this, the regression model (coded) is given by Equation 3.

$$Hardness = +1945.25472 - 2.98294 * X1 - 1.37764 * X2 - 4.50525 * X3 - 7.20533E - 003 * X1 * X3 + 5.21970E - 003 * X1^2 + 5.06749E - 003 * X2^2 + 0.010594 * X3^2 \quad (3)$$

where X_1, X_2 and X_3 are the code values of the test variables (heating temperature, holding time, and stabilisation temperature).

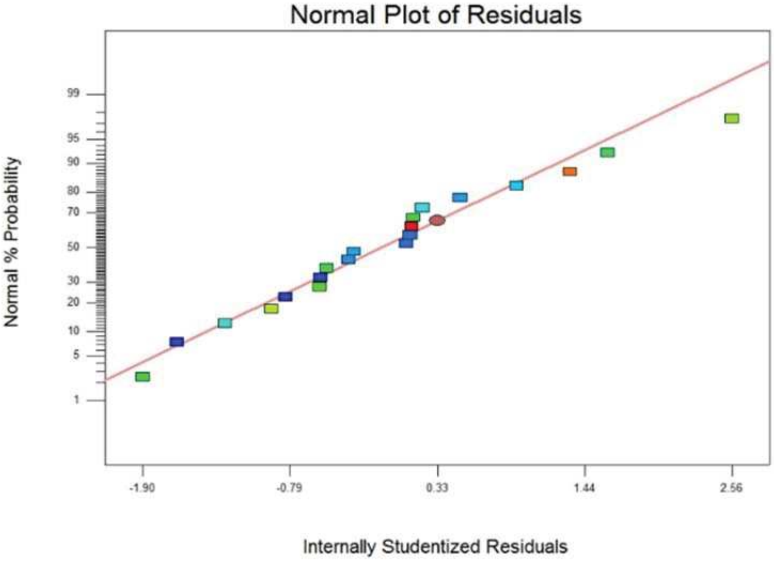

Fig. 3. Normal probability plot of residual

Figure 4 presents the response surface considering interaction among the independent variable. As can be seen from Figure 4, when both heating temperature and holding time is at the low level, the hardness value is at high value. The corresponding perturbation chart of Figure 5 is used to understand the sensitivity of the process variable toward the hardness property of the aluminium foam [18]. From the figure, it can be deduced that the dependence of hardness characteristic with both heating temperature and holding time is nonlinear. The rate of decrease in hardness is high when the heating temperature and holding time is added, commencing from -1 to -0.5. The hardness increase as the stabilization temperature is increased from 0.5 to 1. The dependence of the hardness and stabilization temperature is also nonlinear. The hardness decreases with the holding time addition. Therefore, the highest value of hardness is obtained when both the heating temperature and holding time is at low level and the stabilization temperature at the high level.

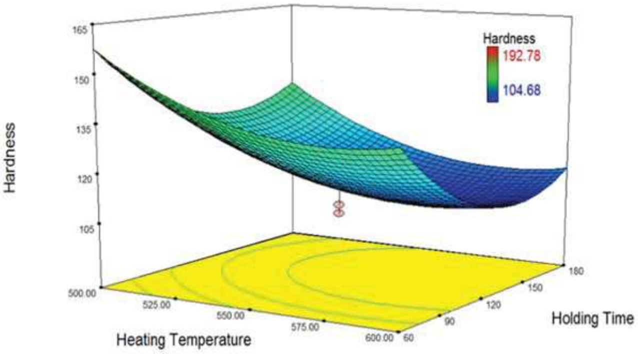

Fig. 4. The response surface estimated from the empirical model of CCD

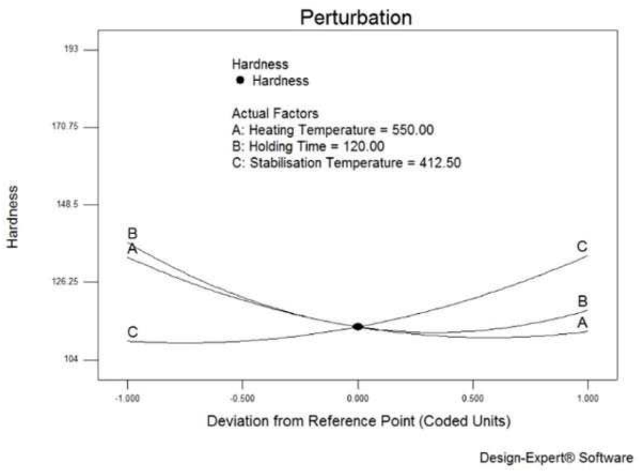

Fig. 5. Perturbation plot for hardness

3.2. Prediction and optimization

At the optimization step, the selection of optimum value of the response variables were generated from regression model using software's numerical optimization algorithm [14]. The predicted and verification test result of the hardness is shown in Table 3. The verification test result was found to be close to the prediction. It is obvious that the high value of hardness is obtained from the low level of both heating temperature and holding time, and high level of stabilization temperature. These results are in agreement with the previous study done by Jeenager [20] in which the high value of microhardness was obtained from low level of holding time. Figure 6 shows the response surface interaction from the independent variable in the optimization.

Table 3.
Predicted and verification test result for hardness

Independent variable			Hardness	
Heating, °C	Holding Time, min	Stabilization temperature, °C	Predicted, HV	Verification Test result, HV
500.2	60.01	450	192.8087	196.68

3.3. Compression testing

Figure 7 shows the compressive strength value of closed cell aluminium foam. It can be seen that the highest value of 2.4 N/mm^2 is obtained by Std 5 sample. The Std 15 sample exhibits compressive strength of 1.85 N/mm^2. The as received and Std 13 sample exhibits compressive strength of 1.6 N/mm^2 and 1.36 N/mm^2 respectively.

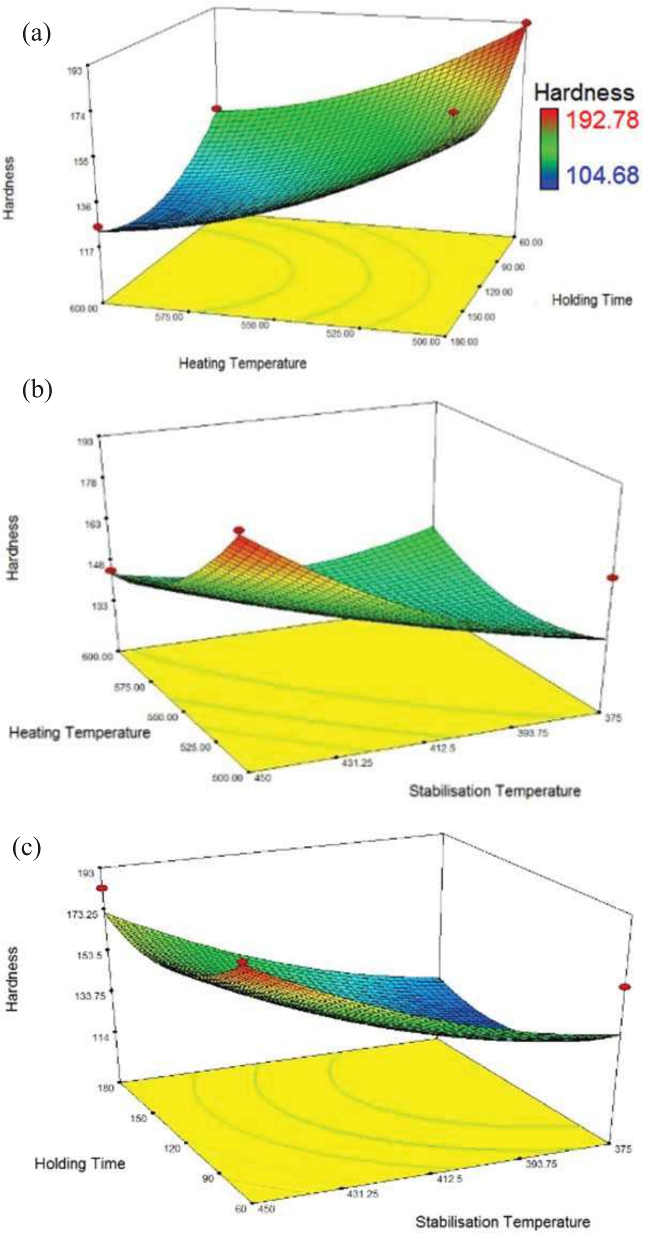

Fig. 6. The response of surface interaction from the independent variable in the optimization: (a) interaction of heating temperature and holding time, (b) interaction of heating and stabilization temperature, and (c) interaction of holding time and stabilization temperature

The compressive stress-strain behaviour of the aluminium foams with different treatment process is presented in Figure 8. The curves are commonly characterized by an initial elastic response, followed by a deformation "plateau" with a positive slop and finally a transition to densification.

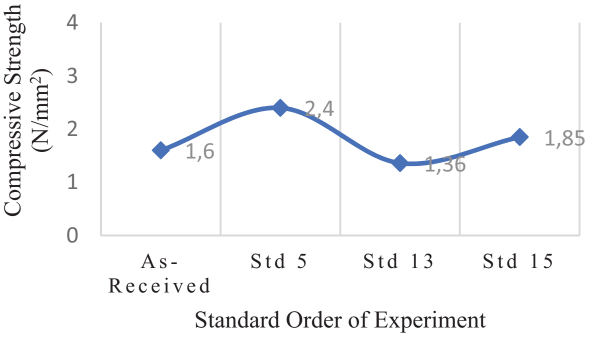

Fig. 7. The compressive strength value of closed cell aluminium foam

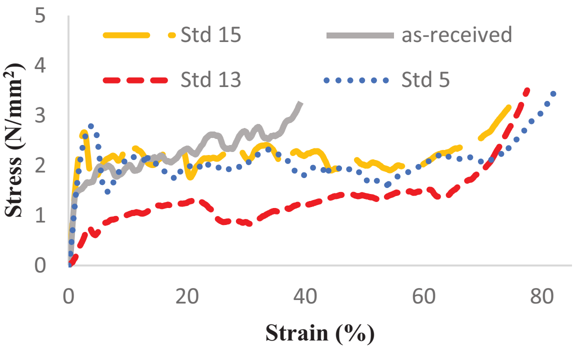

Fig. 8. The compressive stress-strain curves

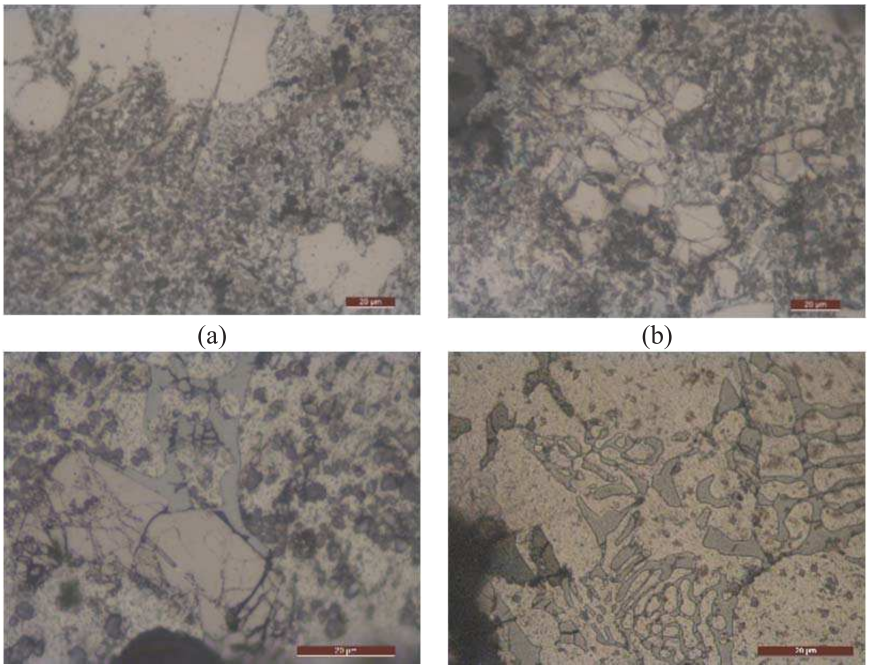
(a) (b) (c) (d)

Fig. 9. The microstructure of closed cell aluminium foam treated by stress relieving method of: (a) as received al foam, (b) high value of hardness, (c) low value of hardness, and (d) cell wall with dendritic growth

Throughout the compression testing, the collapse of the closed cell aluminium foam is observed to progress layer by layer starting at the culmination propagation until full densification appearing [20]. As a result, the load displacement of as received sample is lower than the other. It means that the stress relieving method can be used to improve the mechanical strength of the external wall [21]. From Figure 7, it can be noted that the as-received, Std 5, and Std 15 samples present a jagged curve and stress peak, indicating a brittle fracture. The ductile fracture that occurred in Std 13 sample contributes to the smooth and progressively rising stress-strain behaviour phenomenon [22].

3.4. Microstructure of the material

The mechanical properties of the closed cell aluminium foam such as hardness and compressive strength are affected by the size of the grain and precipitation of foaming agent in the microstructure [17-18]. The microstructure of closed cell aluminium foam treated using stress relieving method is shown in Figure 9. The microstructure of as received aluminium foam is illustrated in Figure 9 (a). It can be seen that, this larger grain size in the material has resulted to a low hardness value of 85.16 HV. When the sample is treated at 500°C heating temperature, holding time of 60 minute, and stabilization temperature of 450°C the sample exhibits a higher hardness value of 192.78 HV. The microstructure with highest value of hardness has small grain size and well distributed intermetallic particle as shown in Figure 9 (b). The sample with the lowest hardness value, however, possesses large grain size and the intermetallic particle in the system is not well distributed as depicted in Figure 9 (c). The material with smaller grain size is harder and stronger than the material with larger grain size. This causes the former has greater total grain boundary area to impede the dislocation motion in the system [23]. The distribution of intermetallic particle is mostly found in the wall boundaries and plateau borders of the cell structure [24]. As illustrated in Figure 9 (d), the dendrites growth from cell wall is noticeable when the sample is treated with both heating temperature and holding time at high level of 600°C and 180 min respectively.

4. Conclusions

In this current work, the influence of stress relieving parameters on the hardness of closed cell aluminium foam have been optimized using central composite design method and ANOVA. The study reveals the highest value of hardness of 192.78 HV was obtained when the stress relieving process is set with the following parameters: heating (500°C); holding time (120 min) and stabilization temperature (450°C). Based on this setting parameters, the closed cell aluminium foam exhibits greater total grain boundary zone that can obstruct the dislocation motion in the system which contributes to the higher value hardness characteristic of the sample. It is also noted that higher heating temperature and longer holding time will produce sample with larger grain size and has an opposing effect on the hardness value.

Acknowledgements

The authors acknowledge the support provided by Universiti Teknologi PETRONAS and Ministry of Higher Education (MOHE) of Malaysia through Fundamental Research Grant Scheme (FRGS) no. FRGS/1/2015/TK05/UTP/02/4.

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