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The Influence of Particle Sizes and Compaction Pressure on Surface Hardness of Aluminum Composite Fabricated Via Powder Metallurgy

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Abstract: A statistical analysis was used to anticipate the influence of particles sizes and compaction pressure on surface hardness of aluminum composites after compacted in a rigid die under an uniaxial compaction. Al-20 wt.% slag powder mixtures with various particle sizes (38µm to 212µm) were prepared and their compressibility was studied in a wide range of compaction pressure up to 300 MPa. All of compacted specimens were sintered at 500^0C for 2hrs. The surface hardness of each sintered specimens was measured by using Vikers Macrohardness. The outcomes of the statistical analysis are predicted by using linear or nonlinear correlation. A direct correlation between compaction pressure and surface hardness of composites was noticed. The correlation between particle sizes and surface hardness are in positive quadratic relationship. It can be concluded that the particle size and compaction pressure significantly influence the surface hardness of aluminum composite.

Key words: Particle size; Compaction pressure; Surface hardness; Statistical analysis.

INTRODUCTION

Powder metallurgy techniques have several advantageous over wrought or cast techniques in producing products such as composite materials, porous material, refractory metals and special high duty alloys (Selvalumar et. al., 2005; Fillabi *et al*., 2007). Aluminum-based particulate-reinforced composites have been fabricated by using powder metallurgy since several decades. Powder metallurgy techniques are based on the classical blending of matrix powders with reinforcing particulates, compacting and sintering. Several advantageous such as better uniformity of the reinforcement distribution in the metal matrix, less residual voids and dissolved gases in products, almost no reaction between the matrix and ceramic particles, near-net shape forming of compacts, and good dimensional tolerances and mechanical properties can be obtained by powder metallurgy. However, some problems are noticeable during compaction of composite powders compared to unreinforced matrix such as low green density, insufficient strength to support secondary processing like sintering, machining or extrusion (Eksi & Yuzbasoglu, 2005; Hafizpour *et al*., 2009). Previous researchers have considered some factors such as volume fraction, particle size, compaction pressure, thermal conductivity, thermal expansion, hardenability, alloy composition, density, impurities and residuals as well as fabrication and processing history in order to optimum the quality of products (Witherell, 1882; Kurt *et al*., 2000; Hamill, 2001). Some attempts have been performed to develop the compaction equations in order to predict the effect of reinforcement particle size and volume fraction on the compressibility of composite products. Moreover, they found that the empirical fitting constants are essentially required to relate the density to compaction pressure (Hafizpour *et al*., 2009). There are several models such as analytical models, discrete element models and neural network models that have been used by researchers in order to predicts correlation between factors that influence properties of composite (Hafizpour *et al*., 2009). Recently, analytical model by statistical approach was used to investigate the effect of various parameters on wear behavior of composites (Kumar and Balasubramaniam, 2008; Zamri *et al*., 2010a; Zamri *et al*., 2011). They used analysis of variance (ANOVA) in order to identify the significant control factors. Furthermore, they have developed the mathematical model in order to predict wear rate incorporating various factors that influence wear behavior of aluminum composite (Zamri *et al*., 2010b).

In comparison with previous studies, the relationship between hardness with compaction pressure, particle sizes of raw materials and porosity has not been extensively investigated. This work attempts to perform investigation using statistical analyses (SPSS version 13) to justify the influence of particle sizes and compaction pressure on surface hardness of aluminum reinforced with 20 wt. % Slag (palm oil factory waste material) fabricated by powder metallurgy. ANOVA was used to predict the tendency of relationship between particle sizes and compaction pressure to the surface hardness followed by mathematical modeling.

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2.0 Experimental Procedure:

2.1 Materials:

Aluminum powders (±45 µm) as matrix material was supplied by the Eyetech, Penang. Slag particles as reinforcement with mean diameter of 38, 75, 125 and 212 µm were supplied by the Jerangau Palm Oil Factory, Terengganu. Figure 1 shows the particles shape and different size of slag particles whereas Figure 2 shows the aluminum powder in round and irregular shape.

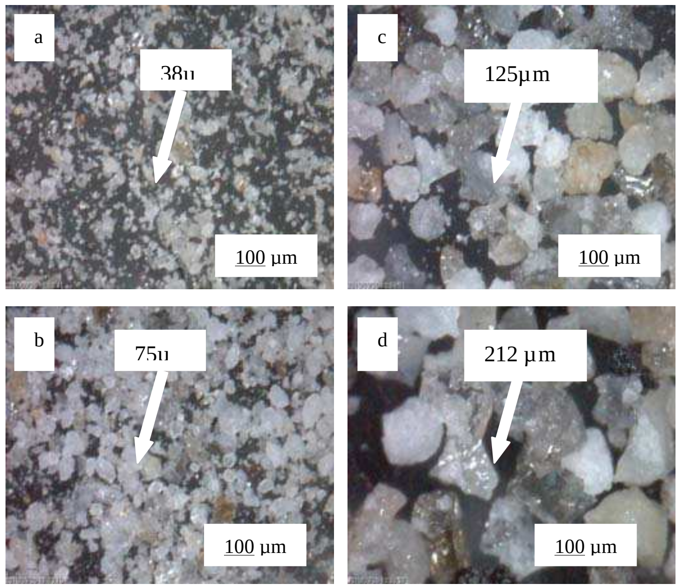

Fig. 1: Optical Micrograph of slag particles at different particle size: (a)38µm; (b)75µm; (c)125µm; (d)212 µm.

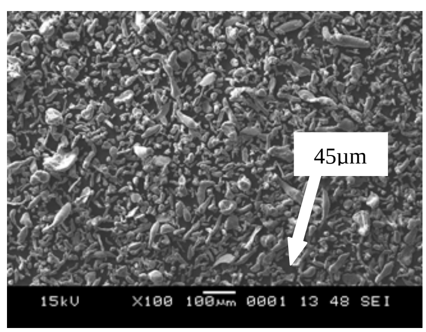

Fig. 2: SEM Micrograph of aluminium powder in irregular and round particle shape at ±45 µm particle size.

2.2 Specimen Preparation:

Aluminium matrix composite reinforced slag was fabricated by powder metallurgy technique. Figure 3 shows the flowchart of the fabrication process. Depending on the reinforcement content, different proportions were prepared as shown in Table 3.6. Powders were mixed by using horizontal mill at 95rpm (diameter container 125 mm) for 10 minutes of mixing time without ball and control agent in order to prevent agglomerated of mix powder that due to heat from collisions of ball mill. The compaction method was uni-axial compacting using the Universal Testing Machine. The pressure used was 200 MPa. The specimens were cold pressed in a die (as shown in Figure 4) to produce a pin of 8 mm in diameter and 10 - 12 mm in height.

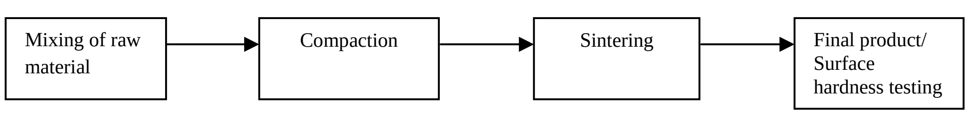

Fig. 3: Flow chart of the process.

Figure 4 shows the die used for compaction of the powder into a pin. The aluminum powders fill in the cavity of the die and followed by cold pressed with a plunger at 150, 200, 250 and 300 MPa. The aluminum powder was pressed into a pin of length 10 - 12 mm with a flat surface of 8 mm in diameter at the both ends as shown in Figure 5.

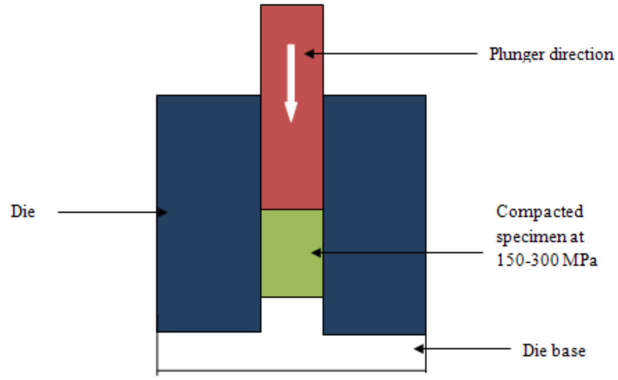

Fig. 4: Schematic diagram of the uni-axial compaction process.

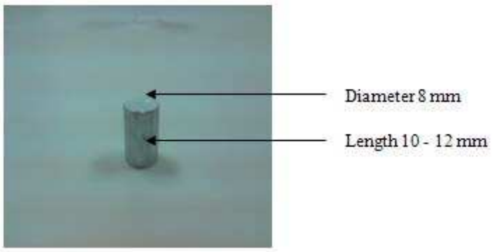

Fig. 5: A typical compacted pin after compaction process.

Figure 6 shows all specimens are arranged properly in the carbolite furnace chamber according to the coded that has been labeled before. After that, the carbolite furnace door has been closed and the sintering temperature and time have been setting up by using control panel at the carbolite furnace. All compacted pin were sintered under different sintering temperatures at fixed sintering time and temperature. Figure 7 shows one example of sintering stage described by graph temperature versus time during sintered under 500 ^{0}C temperature at 15 ^{0}C/ min for 2 hrs.

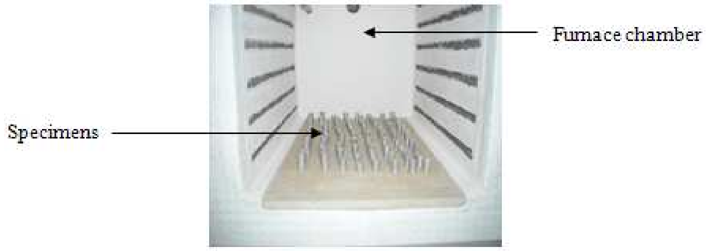

Fig. 6: All compacted specimens are located in the carbolite furnace for sintering process.

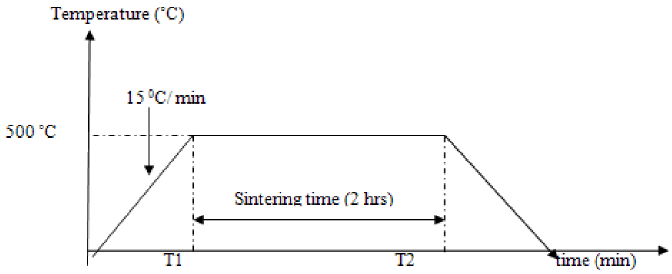

Fig. 7: Temperature versus time in setting up the sintering process.

2.3 Hardness Measurement:

In order to study the changes of hardness with increasing reinforcement content, the hardness measurement (macrohardness) has been conducted using the indentation method with a Vickers hardness tester (Mitutoyo) according to equation 1. The hardness measurements of the specimens were carried out using a 136^0 Vickers diamond pyramid indenter with a load of 9.807 N and a dwell time of 10 seconds. The load speed was 20µm/s. The specimens were fine ground with 600 grit abrasive paper before testing. The result was obtained from an average of 5 measurements at different location for each specimen.

$$\text{Vikers Hardness Value (VHN)} = \frac{1.854\,P}{D^2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

Where P = applied load, kg
D = distance between two opposite corner, mm.

2.4 Microstructure Evaluation:

To investigate the microstructure of the specimens, the microstructure of the monolithic matrix and composites was examined by digital optical microscope. Specimens for microscopic examination were prepared by standard metallographic procedure and then etched with Keller's reagent.

RESULTS AND DISCUSSION

The Vickers hardness test was conducted as per the conditions dictated by the design matrix (Table 1) using Vickers Hardness Tester (Mitutoyo Japan). Ten measurements are taken at different location on two end surfaces of each specimen. The surface hardness of each measurement was recorded and presented in Table 1.

Table 1: Particle sizes and compaction pressure for each specimen and Vickers hardness experimental results (No.= Specimen number, CP= Compaction Pressure, PS= Particle Size, SH= Surface hardness).

No.	CP	PS	SH	No.	CP	PS	SH	No.	CP	PS	SH	No.	CP	PS	SH
1	150	0	148	41	200	0	196	81	250	0	218	121	300	0	279
2	150	0	175	42	200	0	213	82	250	0	223	122	300	0	305
3	150	0	180	43	200	0	208	83	250	0	229	123	300	0	272
4	150	0	174	44	200	0	278	84	250	0	226	124	300	0	275
5	150	0	188	45	200	0	202	85	250	0	232	125	300	0	236
6	150	0	152	46	200	0	192	86	250	0	214	126	300	0	281
7	150	0	150	47	200	0	189	87	250	0	211	127	300	0	297
8	150	0	155	48	200	0	199	88	250	0	234	128	300	0	276
9	150	0	163	49	200	0	220	89	250	0	256	129	300	0	312
10	150	0	165	50	200	0	237	90	250	0	249	130	300	0	302
11	150	38	122	51	200	38	150	91	250	38	92	131	300	38	202
12	150	38	126	52	200	38	132	92	250	38	176	132	300	38	146
13	150	38	111	53	200	38	136	93	250	38	125	133	300	38	206
14	150	38	125	54	200	38	154	94	250	38	164	134	300	38	242
15	150	38	124	55	200	38	159	95	250	38	128	135	300	38	162
16	150	38	122	56	200	38	120	96	250	38	118	136	300	38	223
17	150	38	126	57	200	38	128	97	250	38	118	137	300	38	206
18	150	38	111	58	200	38	155	98	250	38	159	136	300	38	183
19	150	38	125	59	200	38	166	99	250	38	185	139	300	38	177
20	150	38	124	60	200	38	146	100	250	38	154	140	300	38	175
21	150	75	124	61	200	75	130	101	250	75	145	141	300	75	156
22	150	75	121	62	200	75	112	102	250	75	127	142	300	75	134
23	150	75	155	63	200	75	106	103	250	75	127	143	300	75	119
24	150	75	140	64	200	75	120	104	250	75	117	144	300	75	149
25	150	75	154	65	200	75	122	105	250	75	113	145	300	75	138
26	150	75	86	66	200	75	134	106	250	75	135	146	300	75	179
27	150	75	130	67	200	75	111	107	250	75	146	147	300	75	186
28	150	75	92	68	200	75	122	108	250	75	182	148	300	75	194
29	150	75	145	69	200	75	112	109	250	75	131	149	300	75	164
30	150	75	126	70	200	75	129	110	250	75	162	150	300	75	207
31	150	212	120	71	200	212	156	111	250	212	214	151	300	212	222
32	150	212	120	72	200	212	142	112	250	212	216	152	300	212	182
33	150	212	97	73	200	212	271	113	250	212	214	153	300	212	268
34	150	212	154	74	200	212	166	114	250	212	156	154	300	212	212
35	150	212	170	75	200	212	181	115	250	212	229	155	300	212	230
36	150	212	113	76	200	212	108	116	250	212	142	156	300	212	208
37	150	212	131	77	200	212	177	117	250	212	158	157	300	212	215
38	150	212	124	78	200	212	204	118	250	212	189	158	300	212	328
39	150	212	106	79	200	212	111	119	250	212	169	159	300	212	216
40	150	212	135	80	200	212	166	120	250	212	298	160	300	212	303

Dependent variable: Surface hardness (SH),
Independent variable: Compaction pressure (CP), Particle size (PS)

3.1Correlation Between Compaction Pressures With Surface Hardness:

The measured macrohardness of Al-20 wt. % slag composite at different compaction pressure is presented Figure 8. From the graph, it is evident that the higher compaction pressure gives the higher the surface hardness. In order to investigate the degree of influence of compaction pressure to the surface hardness, the results are treated based on statistical analysis of average and analysis of variance (ANOVA).

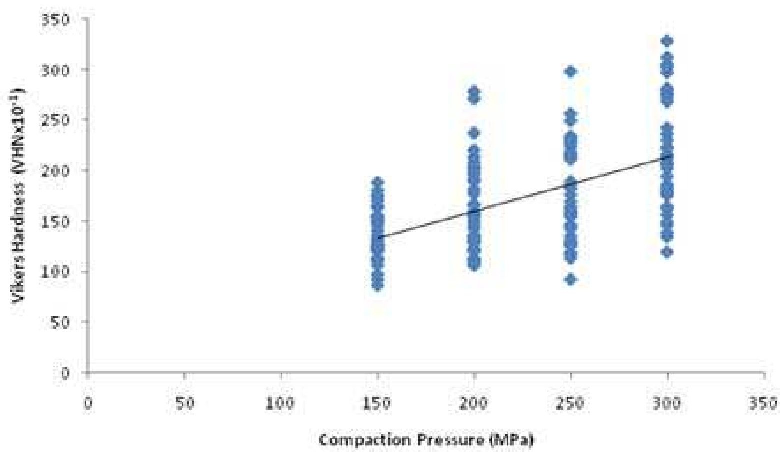

Fig. 8: Correlation between surface hardness and compaction pressure for all specimens.

Table 2 shows the statistical analysis significant data for the surface hardness results. Based on the statistical analysis result (Table 2), there are strong positive linear correlation of compaction pressure on the surface hardness (P.Corr = 0.555, Sig = 0.000, whereby the analysis carried out for significant level 1%). The contribution of compaction pressure on the surface hardness has been justified by regression analysis as indicated in Table 2. It was found that the compaction pressure shows the significant contribution expressed by F = 70.51 and Sig = 0.000. It also shows that positive Beta value (Beta = 0.555, Sig = 0.000) whereby can justify that 1 MPa compaction pressure increased 0.555 HVN surface hardness of the composite. The compaction pressure contributes to the surface hardness is 30.4% as shown in Table 2 (P (%) = 30.4).

Table 2: Statistical analysis significant data for surface hardness result.

Variable	Adj. R^2	Std. Error	Beta	Df	P.Corr	F	Sig.	*P* (%)[#]
[a]CP	0.304	45.03	0.555	159	0.555[**]	70.51	0.000	30.4

Method: enter (All requested variables entered),
Dependent variable: Surface hardness (SH),
[a]Independent variable: Compaction pressure (CP),
[#]Contribution percentage of independent variable
**Correlation is significant at the level 0.01 level (2-tailed)

Therefore, based on statistical result, the correlation between compaction pressure and surface hardness is significantly in linear correlation. It can be expressed in mathematical model as:

$$SH = 0.555CP \qquad (2)$$

The compaction pressure is believed to control the porosity of powder metallurgy product. The optical micrographs of the as sintered aluminum composites with 20 wt. % slag, using different compaction pressures, with various amounts of porosity are shown in Figure 9. Inspection of the sintered porosity in the aluminum alloy and composite matrices indicated that no interconnected porosity was present in the matrix alloy, and only closed isolated porosity was observed.

The apparent porosity in the composites as function of the compaction pressure is shown in Figure 10. It is clearly shown that the porosity of the specimens decreased rapidly as the compaction pressure increased from 150 to 300 MPa.

Figure 11 shows the result of the influence of apparent porosity on the Vikers macrohardness of the specimens. The surface hardness of the specimens decreased as the sintered porosity increased.

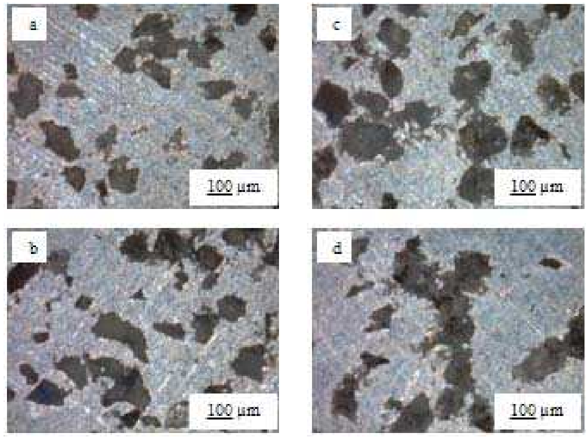

Fig. 9: Micrograph of typical specimens of Al/20 wt. % slag75µm at different compaction pressure (a) 150 MPa (b) 200 MPa (c) 250 MPa (d) 300 Mpa.

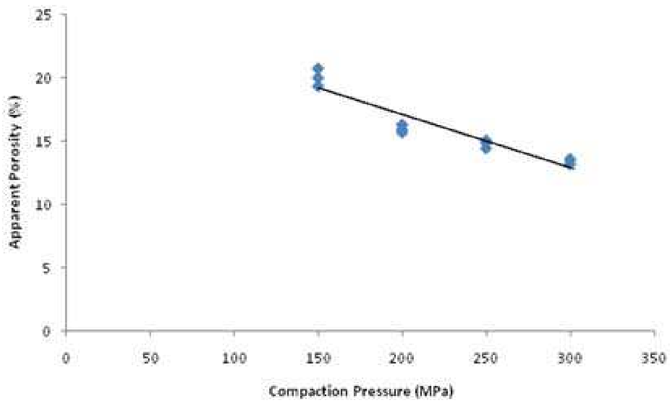

Fig. 10: Variation in apparent porosity in the aluminum composite 20 wt. % slag composite as function of compaction pressure.

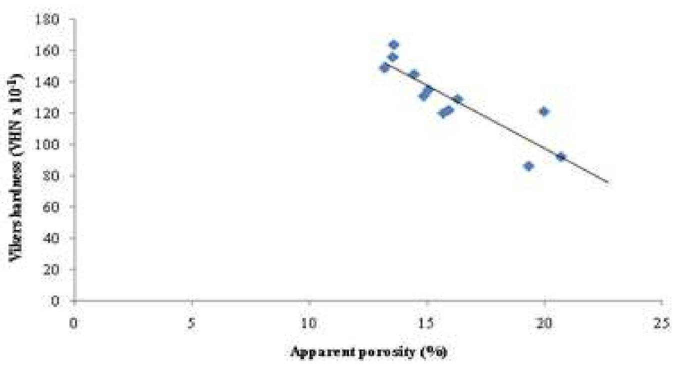

Fig. 11: Vikers hardness versus percentage of apparent porosity in the specimens at different compaction pressure 150 to 300 MPa.

Based on the above results, it is seen that the porosity is clearly affect the surface hardness and well agree with previous researchers that concluded the distinguishing feature of powder metallurgy products is their porosity. According to Dubrujeaud *et al*. (1994) total porosity volume fraction may range 30% for filter or bearing applications down less than 5 % for high-pressure compacted or double press materials. Sinter forging or HIP treatments can lead to even lower porosities (<2%). The presence of porosity negatively influences the mechanical properties of the materials. Tensile and fatigue strengths, hardness, fracture toughness, Young's modulus and elongation (%) decreased with increasing porosity. This reduction in properties is not only influenced by the total porosity vol. %, but also by pore size and shape, as well as pore interconnectivity (Dubrujeaud *et al*., 1994).

3.2 Correlation Between Particle Sizes With Surface Hardness:

Figure 8 shows optical micrographs of the specimens that reveal the distribution of slag particle in the matrix aluminum at for different particle size of slag.

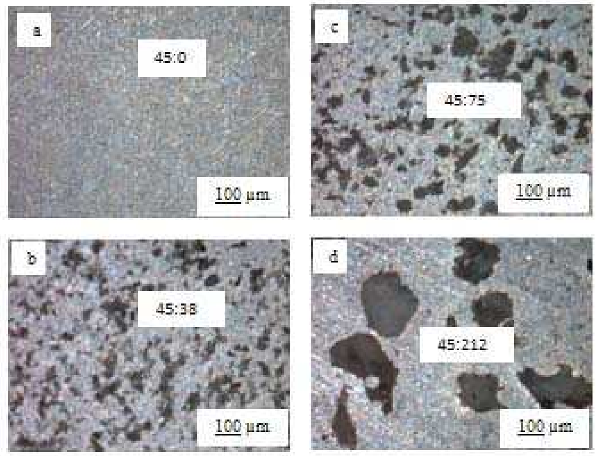

Fig. 12: Micrograph of typical specimens of (a) monolithic matrix Al (b) Al/20 wt. % slag38µm (c) Al/20 wt. % slag75µm (d) Al/20 wt. %slag212µm.

In this work, the ratio of particle size of Al: slag is different at 45:0, 45:38, 45:75, and 45:212. Based on the experimental results as shown in Figure 9, the surface hardness of composites decreased when the particle size ratio are large (45:38 and 45:75), but increased when the particle size ratio is smaller (45:212). Specifically, this trend is due to the relative improvement in surface hardness depends on the particle size ratio of matrix and reinforcement. Within a limit range, the greater the size ratio, the higher the maximum packing density tends to improve surface hardness. This result supported by facts that particle size distribution is one factor that influences the density of powder metallurgy product during compaction stage and bimodal particle blends can pack to higher densities than mono sized particles. (German, 1994).

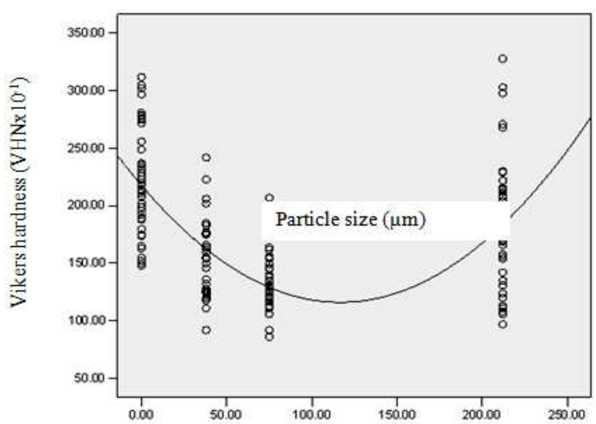

Fig. 13: Correlation between surface hardness and particles size for all specimens.

In order to investigate the degree of particles size influence to the surface hardness, the results have been treated based on regression. During diagnostic plot, it is found that the regression model is suitable to choose as an empirical model in order to anticipate the influence of particles size on the surface hardness. Table 2 shows the regression result by estimation theory, where b_0 (constant), b_1 and b_2 are estimated by the data.

Table 2: Statistical analysis significant data for surface hardness result.

	Model Summary					Parameter Estimates		
Equation	R Square	F	df1	df2	Sig.	Constant	b1	b2
Quardratic	0.359	43.922	2	157	0.000	217.789	-1.741	0.007

Dependent variable: Surface hardness (SH).
Independent variable: Particle size (PS).

Therefore, based on the statistical result, the correlation between particle size and surface hardness is significantly in quadratic correlation that can be expressed in mathematical regression model as:

$$SH = 0.07PS^2 - 1.741PS + 217.789 \qquad (3)$$

Conclusion:

Experimental results showed that it was feasible to prepare aluminum composite reinforced with 20 wt. % slag through powder metallurgy. Various compaction pressure and slag particles size used appeared to have significant effects on the surface hardness of aluminum composite. It was confirmed by statistical analysis result. It was direct correlation between compaction pressure and surface hardness of composite. However, the correlation between particle sizes and surface hardness are in positive quadratic relationship. In summary, the surface hardness of aluminum composite reinforced with 20 wt. % slag is influenced by pressure compaction and particles size.

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