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Optimizing warm compaction parameters on the porosity and hardness of Bronze/Tin ore waste composites

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Abstract. Bronze has widely use in industry as a material for gear, propeller, and others. These products can be produced by powder metallurgy technique via compacting and sintering process. To improve green strength of the green compacted can be done with the warm compaction process. This work aimed to study the optimalization warm compaction parameters such as compacting temperature, compacting pressure, particle size, and particle content when the porosity and hardness of tin ore waste reinforced bronze matrix composites are simultaneous considered. The combination of signal to noise ratio approach in Taguchi method and grey relational analysis was used to evaluate multi responses of composites. The quality characteristic of responses are smaller the better and larger the better. It was found that, the optimal parameter setting is compacting pressure factor at 400 Mpa, compacting temperature is at 350°C, particle size is at 80 µm, and particle content is at 12 % weight.

Keywords: optimizing, warm compaction parameters, porosity, hardness, ore waste

1. Introduction

Copper and it is alloys including bronze have moderate hardness, good thermal conductivity, and good tribological properties. These materials can be used in many application involving bearings, seals, shafts, gears, propellers, etc [1]. These components can be produced in various ways such as casting and powder metallurgy. Powder metallurgy is the most common technology to produce porous material, where the porosity of pores is controlled.

To reduce the cost, which arises from feedstock and manufacturing process has been the main thrust for the research and development of powder metallurgy [2]. Warm compaction is an established technique in powder metallurgy to improve metal powder properties. The experimental results show that with warm compaction porosity lower than 2% [3].

An investigation on the effect of process parameters with powder metallurgy methods such as the effect of compacting on porosity [4] and density [5] has been studied but not simultaneously. Optimalization of the materials and processes for obtaining metal matrix composites usable in industry is a very important thing to do [6].

The aim of this work is made for optimizing compaction parameters of tin ore waste strengthened bronze matrix composites. The experimental design prepared by using orthogonal array based on Taguchi method. Grey relational analysis choosen to optimalization of compacting process



parameters in order to minimize the porosity, as well as to maximize the hardness simultaneously. The specimen has been prepared by using conventional warm compaction pressing.

2. Materials, Methods and Equipments

In this research, tin ore waste strengthened bronze matrix composites were produced by powder metallurgy technique. Raw materials were provided by PT. Timah (Indonesia). The compound was determined using X-ray fluorescence spectrometer (XRF) and X-ray diffraction (XRD). The composition of the mineral consists of zircon (ZrSiO_4) = 75%, cassiterite (SnO_2) = 12%, ilmenite (FeTiO_3) = 9%, and pyrite (FeS_2) = 4%. The XRD analysis supports these results and that pattern is shown in Figure 1. SEM image of zircon particle is given in Figure 2.

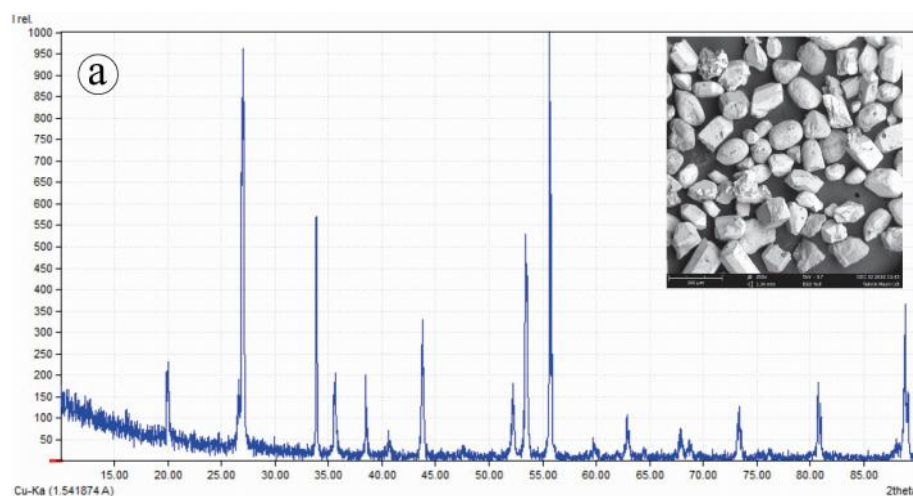


Figure 1. XRD pattern analysis of the starting powder

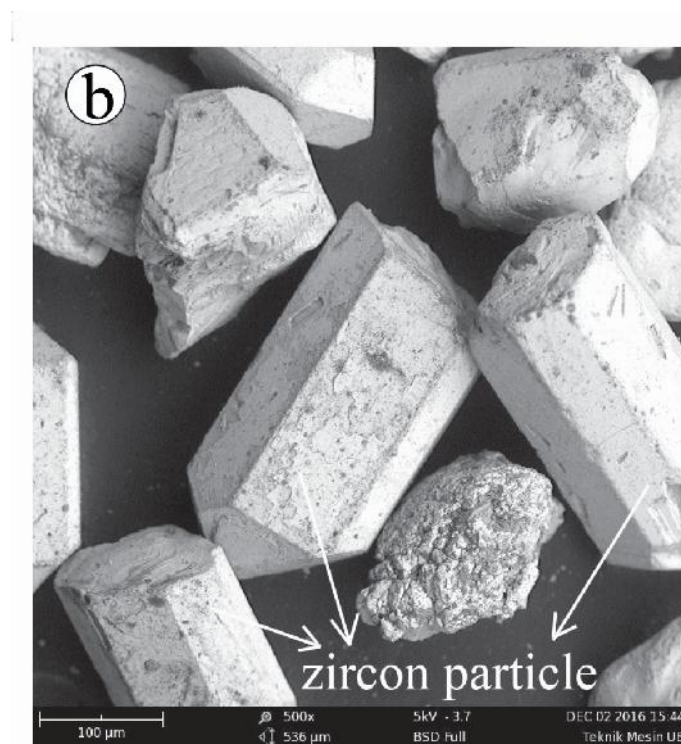


Figure 2. SEM image of zircon sand

The average size of the zircon sand was $D_{50}=133\ \mu\text{m}$. Particle size distribution of minerals is given in Figure 3. In addition, the particle diameter at 10% (D_{10}) is $98\ \mu\text{m}$ and the diameter at 90% (D_{90}) is $194\ \mu\text{m}$. The size of zircon minerals was measured using Particle Size Analyzer (PSA-Cilas 1090 Dry).

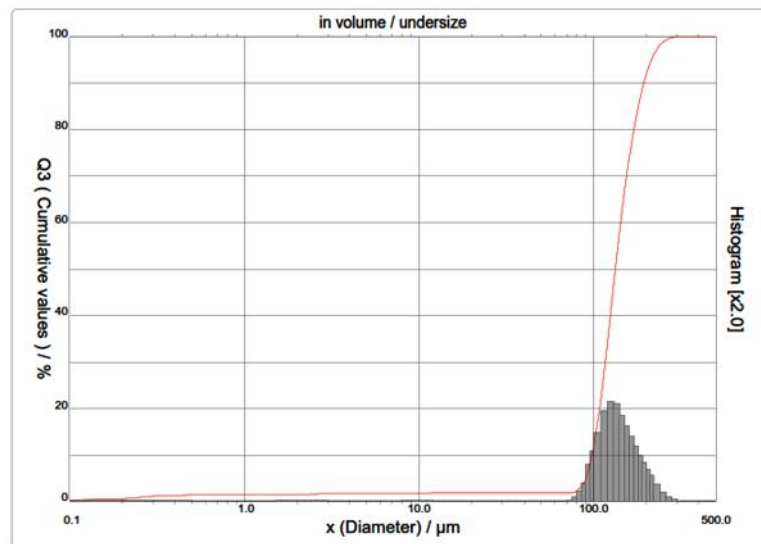


Figure 3. Particle Size Distribution of zircon sand

The chemical composition of starting material was determined using X-ray fluorescence (XRF) spectrometer (PANalytical, Type: Minipal 4) and that is shown in Table 1.

Table 1. The typical chemical composition (%) of zircon sand

Zr	Sn	Fe	Si	Ce	Hf	S	Ti	Ti	Y	Nd	Ni	Ca	another
84.00	4.62	2.38	1.84	1.59	1.28	1.15	1.08	1.08	0.74	0.36	0.24	0.24	balance

To investigation and optimalization warm compaction parameters, nine variation specimens were pressed in two directions by conventional hydraulic press. Zircon particles were added 8%, 10 %, and 12% weight. Table 2 shows the experimental parameters and levels used in this study.

Table 2. Experimental parameters and levels

Parameter	Code	Unit	Level		
			1	2	3
Pressure	P	MPa	300	350	400
Temperature	T	$^{\circ}\text{C}$	300	400	500
Particle Size	PS	μm	80	100	180
Particle Content	PC	% weight	8	10	12

The powders were mixed for 10 minutes. The mixtures were put in the mold. The mold was heated to a temperature variation between $300\ ^{\circ}\text{C}$ to $500\ ^{\circ}\text{C}$. After the heating process, the mixtures were compacted in two directions by using conventional hydraulic press under 300 MPa, 350 MPa, and 400 MPa. The dimensions of the green compact were 40 mm for the outer diameter, 18 mm for

the inner diameter and 7 mm thickness. Schematic of the hydraulic press with heater system and process used in this work is shown in Figure 4 and Figure 5.

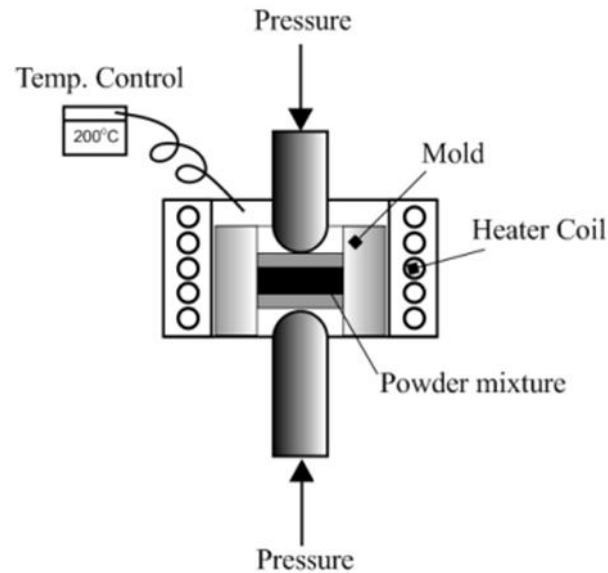


Figure 4. Schematic of the powder press process



Figure 5. Powder compacted process

After sintering process, the green samples were tested to identify the density. The experimental densities were evaluated by using the following formula:

$$\rho_{\text{exp}} = \frac{W_a}{W_a - W_f} \times \rho_f \quad (1)$$

Where, W_a is specimen weight in the air, W_f is specimen weight in fluid (distilled water used in this work), and ρ_f is density of fluid.

The next step was determine the solution of theoretical density of green samples using the mass fractions and densities of each chemical element. The theoretical density was evaluated by using the following equation:

$$\rho_{Th} = \%A_1 \cdot \rho A_1 + \%A_2 \cdot \rho A_2 + \dots + \%A_n \cdot \rho A_n \quad (2)$$

Where, %A and ...A both mass fraction and density of chemical elements

The porosity of green samples was evaluated by Archimedes law. Porosity quantity test used ASTM Standard B311-93, which is the ratio of the green sample difference density (theoretical and experimental) and green sample theoretical density by 100%. The percent porosity of the green sample used this formula:

$$\% \text{ Porosity} = \frac{\rho_{Th} - \rho_{exp}}{\rho_{Th}} \times 100\% \quad (3)$$

3. Taguchi Method

Single response analysis (SRA) is an analysed when there can be only one response quality characteristic. Taguchi technique is a statistical method used to optimize the single response analysis [7]. Taguchi method which combine the experiment design theory and the quality loss fuction concept have been used in developing robust design of processes and products.

3.1. Degree of Freedom (DoF)

To conduct the experiments, the orthogonal array size is determined by calculating degree of freedom of the factors within this range of work. This type of level of factors which is unequally distributed can be tailored to fit the mixed levels of factors and its DoF must be equal or exceed the sum of DoF of all the factors of experiment [8].

DoF is defined as the number of comparisons made between process parameters that are made to determine the better level. That is used to compute the minimum number of experiments that will be conducted to investigate the observed factors. The DoF value is given by equation

$$\text{Degree of Freedom} = \text{number of levels} - 1 \quad (4)$$

The total DoF of experimental factors in this study is given in Table 3.

Table 3. DoF of Experimental factors

Factor	DoF (Level-1)	Sub Total
Pressure	3-1	2
Temperature	3-1	2
Particle Size	3-1	2
Particle Content	3-1	2
Total DoF		8

Because of DoF of orthogonal array size must be equal or exceed the sum of DoF of experimental factors, thus an L9 Taguchi orthogonal array is selected. The DoF of an L9 Taguchi array is $9-1=8$ DoF.

3.2. Orthogonal Array

An orthogonal array is a fractional factorial matrix which assures a balanced comparison of levels of any factor or interaction of factors. The array is called orthogonal because all columns can be evaluated independently of one another [9].

Table 4 shows an orthogonal array $L_9(3^4)$. L_9 is the maximum number of rows (experiments), 3 is the number of levels, and 4 is the number of column (factors). That is used can be used for degree of freedom in Tabel 3 before.

Table 4. Orthogonal array $L_9(3^4)$

Experiment number	Column number			
	1	2	3	4
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	2
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

Based on orthogonal array $L_9(3^4)$ as shown in Table 4 and experimental parameters in Table 2, the experimental layout and the responses of experiment were shown in Table 5.

Table 5. Experimental Layout and Responses

Run	Pressure (MPa)	Temperature ($^{\circ}\text{C}$)	Particle Size (μm)	Particle Cont (% weight)	Porosity (%)	Hardness (Hv)
1	300	350	80	8	46.71	65.11
2	300	400	100	10	37.80	74.33
3	300	500	180	12	37.23	91.11
4	350	350	100	12	44.13	76.18
5	350	400	180	8	37.24	78.65
6	350	500	80	10	36.16	96.29
7	400	350	180	10	38.55	117.37
8	400	400	80	12	37.57	129.67
9	400	500	100	8	40.86	102.07

3.3. S/N Ratio

Signal to noise is a quality in the communication and engineering field, where is the ratio between the power of the signal (μ^2) and the power of the noise (σ^2) which expresses in decibels (dB). Taguchi, whose background is communication and electronic engineering, introduced this same concept into the design of experiments to improvement of quality via variability reduction and the improvement of measurement [9]. S/N ratio has been used as the quality characteristics, which available depending on the type of characteristic such as smaller the better, larger the better, or nominal is the best.

Taguchi recommends using the common logarithm of S/N ratio multiplied by 10. Hence, for the case of smaller the better, which is also measured in decibels, the S/N ratio may be calculated as

$$SN = -10\log\left(\frac{1}{n}\sum_{i=1}^n y^2\right) \quad (5)$$

Meanwhile for the larger the better performance quality characteristic, the S/N ratio is defined as

$$SN = -10\log\left(\frac{1}{n}\sum_{i=1}^n \frac{1}{y_i^2}\right) \quad (6)$$

Where n is number of repetitions of the experiment and y_i is observed response value.

Table 6. S/N Ratio for Porosity and Hardness

Run	Porosity S/N Ratio (dB)	Hardness S/N Ratio (dB)
1	-33.388	36.273
2	-31.550	37.423
3	-31.418	39.191
4	-32.895	37.637
5	-31.420	37.914
6	-31.165	39.672
7	-31.720	41.391
8	-31.497	42.257
9	-32.226	40.178

The green compact specimens is shown in Figure 6.



Figure 6. Image of green compacted

4. Grey Relation Analysis

Multiple response analysis (MRA) is a frequency analysed when there can be more than one response quality characteristics. To optimization of MRA can be done using a Grey Relational Analysis (GRA) [10]. It is carried out in step by step stage until the Grey Relation Grade (GRG) is obtained. The GRG value reflect the single optimization value of the multiple performance [11].

The GRG is a helpful method to analyze multiple factors and correlation between sequences with smallest data. It used to describe the score of connection in among two sequences to calculate the gap between two factors [12]. In the Grey relational generating process the responses are normalized in the range of 0 and 1 by using the higher the better condition [10].

The GRG primarily generates a data preprocessing to normalize the S/N ratio data in Taguchi method. That is linearly normalized or grey-relational generating in the range between 0 and 1 [13]. A normalize the S/N ratio of larger the better can be expressed as:

$$x_i(k) = \frac{\eta_i(k) - \min(\eta_i(k))}{\max(\eta_i(k)) - \min(\eta_i(k))} \quad (7)$$

Where $\eta_i(k)$ is the S/N ratio value; $\max \eta_i(k)$ is the largest value of $\eta_i(k)$ for the k^{th} response; $\min \eta_i(k)$ is the smallest value of $\eta_i(k)$ for the k^{th} response.

If the expectancy is smaller the better, then it can be calculated by using the following formula,

$$x_i(k) = \frac{\max(\eta_i(k)) - \eta_i(k)}{\max(\eta_i(k)) - \min(\eta_i(k))} \quad (8)$$

For example, a normalize the S/N ratio for factor hardness (larger the better) based on Tabel 3 can be computed as follows:

$$x_i(1) = \frac{36.273 - 36.273}{42.257 - 36.273} = 0$$

In addition, all of $x_i(k)$ converted into grey relational coefficient or $\xi_i(k)$. The coefficient is calculated to state the relationship among the ideal and the real results of the experiment. That is defined as,

$$\xi_i(k) = \frac{\Delta_{\min} + \xi \times \Delta_{\max}}{\Delta_{oi}(k) + \xi \times \Delta_{\max}} \quad (9)$$

Where $\Delta_{oi}(k)$ is the difference of the absolute value between $x_0(k)$ and $x_i(k)$, $x_0(k)$ is the ideal normalized = 1 [6 khaimovich]; Δ_{\min} is the smallest value of $\Delta_{oi}(k)$; Δ_{\max} is the largest value of $\Delta_{oi}(k)$; and ξ is identification or distinguishing coefficient set in range $0 \leq \xi \leq 1$; it was generally set to 0.5 [14, 15, 16].

For example, grey relational coefficient (GRC) can be calculated as follows:

$$\Delta_{oi}(1) = ||1 - 0|| = 1$$

$$\xi_i(1) = \frac{0 + 0.5 \times 1}{1 + 0.5 \times 1} = 0.333$$

The grey relational grade (GRG) was computed by averaging the GRC or $\xi_i(k)$. It can be defined as follows,

$$\gamma_i = \frac{1}{n} \sum_{k=1}^n \xi_i(k) \quad (10)$$

Table 7 shows the grey relational coefficient for porosity and hardness, grey relational grade, and S/N ratio of GRG.

Table 7. GRC, GRG, and S/N ratio

Run	Porosity GRC	Hardness GRC	GRG	S/N ratio
1	1.000	0.333	0.667	-3.5218
2	0.377	0.382	0.380	-8.4135
3	0.361	0.494	0.427	-7.3849
4	0.693	0.393	0.543	-5.3072
5	0.361	0.408	0.384	-8.3035
6	0.333	0.536	0.435	-7.2323
7	0.400	0.776	0.588	-4.6156
8	0.370	1.000	0.685	-3.2848
9	0.489	0.590	0.539	-5.3608

Table 8. Response table of the average GRG

Factor	Level 1	Level 2	Level 3	Double Average
Pressure	0.4913	0.4540	0.6040*	0.5164
Temperature	0.5993*	0.4830	0.4670	
Particle Size	0.5957*	0.4873	0.4663	
Particle Content	0.5300	0.4677	0.5517*	

* Optimal level

According to the main effects plots for means and S/N ratios shown in Figure 7 and 8, the optimal process parameter can be determined based on the mean value of the S/N ratio. The best parameter setting in this work is the pressure is at 400 Mpa, the temperature is at 350°C, the particle size is at 80 µm, and the particle content is at 12 % weight. Thus indicating that the temperature and particle size can be set at the lowest level, while pressure and particle content can be set at the highest level.

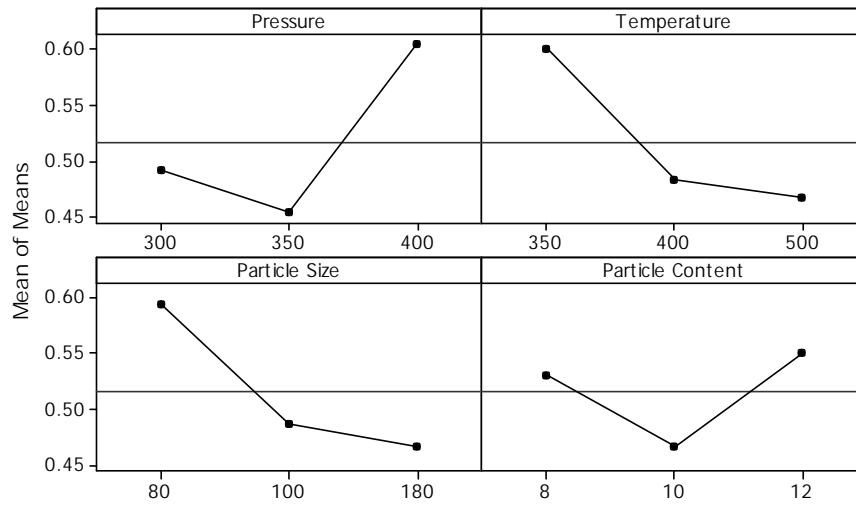


Figure 7. Main effects plots for Means

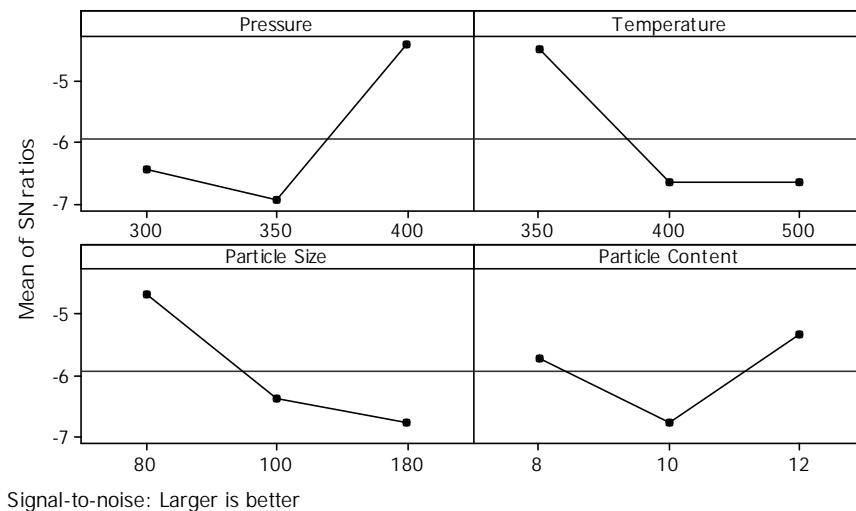


Figure 8. Main effects plots for S/N ratios

ANOVA takes directly the proportion of each sum of squares of the factor effect vector (SS) as the sum of squares of the total variation vector (SS total factor) as a reference of degree of contribution [17]. The analysis of variance for SN ratios is shown in Table 6. Based on the percentage contributions, the pressure exhibited a strong effect in reduce porosity and increase hardness.

Table 9. Analysis of Variance for SN ratios

	DF	SS	MS	% Contribution
Pressure	2	10.7089	5.35445	34.69
Temperature	2	9.5716	4.7858	31.00
Particle Size	2	7.3732	3.6866	23.88
Particle Cont	2	3.2184	1.6092	10.42
Error	*	*	*	
Total	8	30.8721	*	

5. Prediction and Confirmation Test

To predicted GRG value ($\hat{\gamma}$) using the optimal level of the compaction parameters can be calculated as:

$$\hat{\gamma} = \gamma_m + \sum_{i=1}^q (\bar{\gamma}_i - \bar{\gamma}_n) \quad (11)$$

Where, $\bar{\gamma}_n$ the double average of GRG, $\bar{\gamma}_i$ is the average of the GRG at the optimal level, and q is the number of warm compaction parameter that significantly effect the responses.

The predicted GRG value obtained from formula (11) is 0.8015. To verification experiment a confirmation test was conducted. A confirmation test indicates that the optimal values of porosity and hardness are 38.35% and 124.17 Hv, respectively.

6. Conclusion

In this work, Taguchi-orthogonal $L_9(3^4)$ array with grey relational analysis was combined to evaluate multi-response characteristics such as porosity and hardness. Quality characteristic of porosity is smaller the better while the quality of hardness is larger the better. The recommended levels of warm compacting parameters when porosity and hardness are simultaneously considered are the optimum process parameters. The value of parameters are highest compacting pressure (400 MPa), lowest temperature (350°C), lowest particle size (80 μm), and highest particle content (12 wt%).

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