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ENHANCING HARDNESS AND WEAR RESISTANCE OF $ZrSiO_4$-SnO_2/Cu10Sn COMPOSITE PRODUCED BY WARM COMPACTION AND SINTERING

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This research focuses on improving the hardness and wear resistance of $ZrSiO_4$-SnO_2/Cu10Sn composite. In this investigation, the SnO_2 tailing sand dominated by $ZrSiO_4$ and SnO_2 was used for the reinforcement. The Cu10Sn composites with 0, 5, 10, 15 and 20 weight % SnO_2 tailing sand were produced using double action warm compaction followed by sintering. The results show that the highest hardness value and the lowest wear rate of the composites were achieved by adding 15 weight % of SnO_2 tailing sand. Moreover, the wear rate of Cu10Sn matrix composite exhibits a decrease significantly with an increase in the amount of SnO_2 tailing sand due to the presence of hard particles.

Key word: $ZrSiO_4$-SnO_2/Cu10Sn, double warm compaction, sintering, hardness, wear resistance

INTRODUCTION

CuSn is the most common nonferrous metal used for bearing, seal, shaft, wear part, etc. [1, 2]. Lately, this material is often reinforced by adding other matters to increase its strength. Such elements as Al_2O_3, Cr, Ag and volcanic tuff have been reported to improve the durability, strength, hardness, lubricating behavior and wear resistance of copper matrix composites [2-4]. Besides, the mechanical properties of ceramic refractories such as $ZrSiO_4$ reinforced nonferrous metal matrix composites (MMCs) with various techniques to improve the hardness, ultimate tensile strength, and wear resistance have been examined in recent years [5, 6]. Early studies have shown the low friction and wear rate of $ZrSiO_4$ reinforced bronze composites produced by using powder metallurgy (PM) with a single pressed cold compaction method [6].

PM is a traditional forming process generating parts with near shape and without the need of machining process [7]. Further, PM technology refers to high material utilization to reduce the cost of manufacturing process [8], and it can also control the level of composite porosity.

Warm compaction method has been investigated intensively to achieve a higher green compact and sintered densities of metal matrix composites (MMCs) in the last few decades. Warm compaction is a traditional method to improve metal powder compressibility in PM materials [9]. In this method, with the same pressure on cold compaction, the relative density will be higher.

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Recently, researchers have shown interest in using tailing sand as reinforcement in the metal matrix composites industry. Utilizing the tailing sand can reduce production cost and is helpful to solid waste treatment [10]. However, previous researchers have not examined SnO_2 tailing sand dominating $ZrSiO_4$ and SnO_2 strengthened bronze matrix composites. To identify the effect of adding reinforcement at the composite, Cu10Sn matrix composites with a different variation of SnO_2 tailing sand content were produced by warm compaction with double press method. The main objective of this study is to investigate the density, porosity, hardness and wear rate of SnO_2 tailing sand/Cu10Sn composite.

MATERIALS AND METHODS STARTING MATERIALS

The starting materials used for this study were Cu10Sn powder and SnO_2 tailing sand. On average, the particle size of Cu10Sn was 70 mesh. The SnO_2 tailing sand was dominated by $ZrSiO_4$ and SnO_2, respectively. The average size of SnO_2 tailing sand was measured by using particle size distribution analyzer (CILAS 1090 DRY). The diameter at 50 % (D50) of SnO_2 tailing sand was 97,7 μm. The phase crystallite of SnO_2 tailing sand which is shown in Figure 1 (a) was identified using X-Ray Diffraction (XRD, E'xpert Pro - PANalytical) and High Score Plus Software. Also, the morphology of SnO_2 tailing sand, shown in Figure 1 (b), was analyzed by using a scanning electron microscope (SEM, Phenom G2 Pro).

The chemical composition of SnO_2 tailing sand was analyzed using X-ray fluorescence spectrometer (XRF, MiniPAL4, PANalytical). The chemical composition is presented in Table 1.

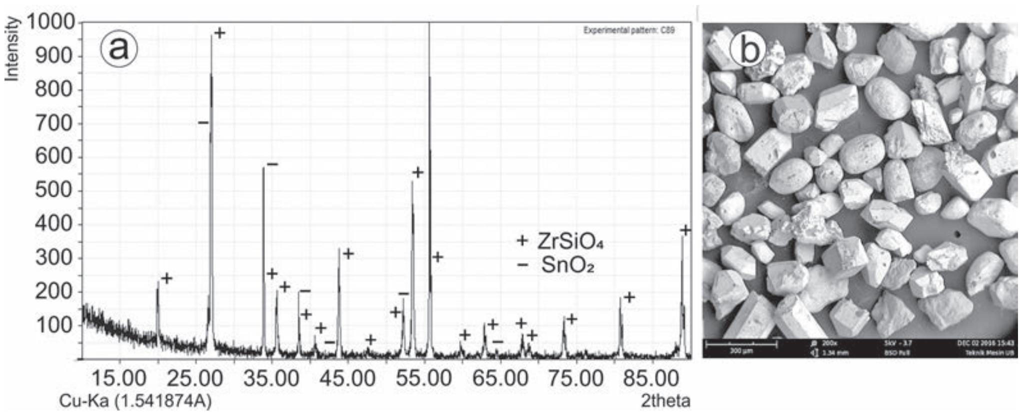

Figure 1 (a) X-Ray Diffraction (XRD) pattern and (b) scanning electron microscope (SEM) Image of SnO_2 tailing sand.

Table 1 **Chemical composition / wt. %**

Zr	Sn	Fe	Si	Ce	Hf
84,00	4,62	2,38	1,84	1,59	1,28
S	Ti	Y	Nd	Ni	Ca
1,15	1,08	0,74	0,36	0,24	0,24
Yb	Pb	Ta	Bi	Th	Other
0,06	0,05	0,12	0,10	0,18	balance

To compare the hardness and wear rate in the composition of the SnO_2 tailings sand/Cu10Sn composites, five different specimens were produced by warm compaction and sintering. The variations in reinforcement contained 0, 5, 10, 15 and 20 % by weight of SnO_2 tailing sand. Replicas were produced three times for each composition.

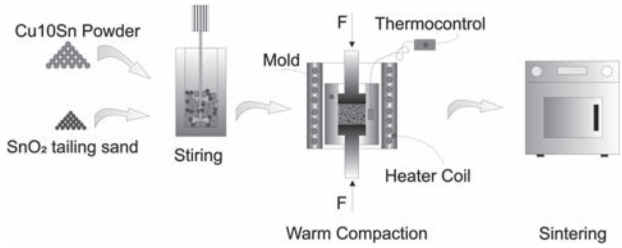

Figure 2 Schematic diagram of the composite production process.

STARTING MATERIAL

In this experimental study, the Cu10Sn powder and SnO_2 tailing sand were mixed using stirrer apparatus for 10 minutes. The mixture was then compacted to produce the green compacts by warm compaction with two directions pressing at a temperature of 300 °C, a pressure level of 48 MPa, and holding time of 20 minutes. The dimensions of the samples were d_{outer} = 40 mm, d_{inner} = 17 mm and thickness = 7 mm. The pressureless sintering of green compacts was carried out in a muffle furnace in an air atmosphere. In the initial phase of the sintering process, the specimens were initially held at a temperature of 400 °C for 20 minutes to remove the fluid attached to the surface of the sample. The sintering temperature was increased gradually to a temperature of 600 °C in 30 minutes and held at this constant temperature for 60 minutes. After that, the heat was reduced until it reached room temperature. Figure 2 presents the schematic diagram of the production process.

The bulk density of specimens was determined according to the Archimedes water immersion method (ASTM Standard B962-14) before and after the sintering process. Distilled water was used to measure sample densities as immersing medium at 25 °C [11].

The porosity value of specimens was calculated in percentage using the Archimedes' principle. The formula used is as follows:

$$\%\ \text{Porosity} = \left(1 - \frac{\rho}{\rho_{th}}\right) \times 100 \qquad (1)$$

where *r* and ρ_{th} are the bulk density and the theoretical density of specimen, respectively.

Rockwell Hardness Tester (Brevetti AFFRI – 206 RT, Italy), with 1/16 inch steel ball as an indenter and 100 Kgf load, was used to measure the hardness of specimens. The microstructure of the samples was observed by using a Scanning electron microscope (SEM, Inspect-S50, FEI, USA).

Composite wear resistance was evaluated using the ogoshi method where the surface of the specimen was plane, and the contact was initially a line with the cylinder. The sizes of the specimen and the cylinder were Æ 40 mm ´ 7 mm and Æ 28 mm x 3 mm, respectively. The sample was made of sintered composites. The cylinder made from hardened steel material with Roughness (Ra) = 4,57 μm and hardness of 25 HRC used as the counterpart. Specimen wear tests were executed at a load of 58,8 N with a constant shear speed of 0,54 ms^{-1}. The wear resistance measurement refers to the method of weight loss. All wear resistance test were executed three times under the same parameters to ensure the repeatability of experimental data. The weight loss was measured by using an electronic digital weighing scale. Based on the weight loss method [12], the wear rate / mm^3m^{-1} was calculated using:

$$\text{Wear rate} = \frac{M}{D \times S} \qquad (2)$$

where *M* represents the weight loss (gram), *D* represents the density / g/mm^3, and *S* represents sliding distance / m.

RESULTS AND DISCUSSION

As shown in Figure 3, the density value slightly decreased with the increasing content of SnO_2 tailing sand in the composites. The decreased was due to the presence of low-density particle as the reinforcement in the composite. This case is similar to that discussed in the literature in terms of the relationship between density and $ZrSiO_4$ content [2, 6]. In this case, the decreased density can be associated with the increased porosity of composites.

Based on Figure 3, the porosity of the SnO_2 tailing sand/Cu10Sn composite was marginally increased by increasing the weight percentage of SnO_2 tailing sand. Also, all of the composites after sintering have the porosity value of less than 26 % as a result of the low-temperature sintering process. In this case, the increased porosity of SnO_2 tailing sand/Cu10Sn composites is due to more voids. Besides, the increase in composite porosity may be related to non-homogeneous powder after the mixing process which results in agglomeration causing poor wettability in the composite. This result shows that composite porosity is strongly related to composite density. However, the decrease in composite density resulted in the increase of composite porosity. Similar porosity behavior has been studied by previous researchers who reported that the higher percentage of porosity might also be related to the weak interfacial bonding between reinforcement and matrix [13].

The Rockwell hardness of composites was observed to increase the weight percentage of SnO_2 tailing sand, as shown in Figure 4. According to the figure, the value of composite hardness showed an increase along with the rise of SnO_2 tailings sand content but decreased significantly at 20 % by weight of SnO_2 sand tailings. An increase in composite hardness occured due to the presence of hard particles. It caused dislocation of line movements in the matrix composite inhibited. The decrease in hardness may be due to agglomeration and nonhomogeneous particle of reinforcement in composites. However, the hardness of composites before and after sintering shows an increase due to the addition of reinforcement particles. Also, two crucial reasons for the improvement in hardness

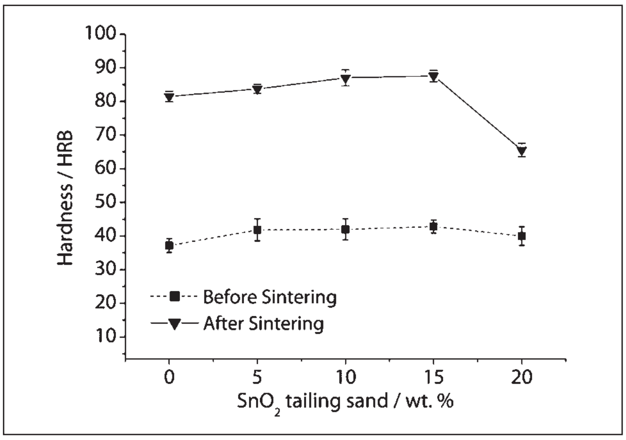

Figure 4 The effect of the SnO_2 tailing sand content on the hardness of composites before and after sintering.

value of particulate reinforced metal matrix composites are particle and dispersion strengthening [14]. Similar to this research work, as a result of particle reinforcement, the addition of hard particles will increase in the value of hardness, as observed by many researchers [13, 14].

The effect of SnO_2 tailing sand amount on the wear rate of the Cu10Sn matrix composite after sintering is presented in Figure 5. The wear rate significantly decreased from 0 to 15 weight % after the addition of SnO_2 tailing sand in Cu10Sn matrix composites. In this case, the wear resistance is proportional to hardness value, as previous studies show [15]. Parallel to the results of density measurement, the SnO_2 tailing sand content decreased the wear rate of SnO_2 tailing sand/ Cu10Sn composites. However, the wear rate value of the composites with 20 % SnO_2 tailing sand started increasing. This case might be due to inhomogeneous particle distribution of the SnO_2 tailing sand particles in Cu10Sn matrix composites, as indicated in Figure 7(e).

Figure 6(a-b) exhibits the SEM micrograph of composite morphology before and after the sintering process. It can be seen that the unreinforced Cu10Sn after sintering (Figure 6(b)) has a smoother structure compared to unreinforced Cu10Sn before sintering (Figure 6(a)). However, the pores of the specimen after sintering were smaller than those before the sintering process.

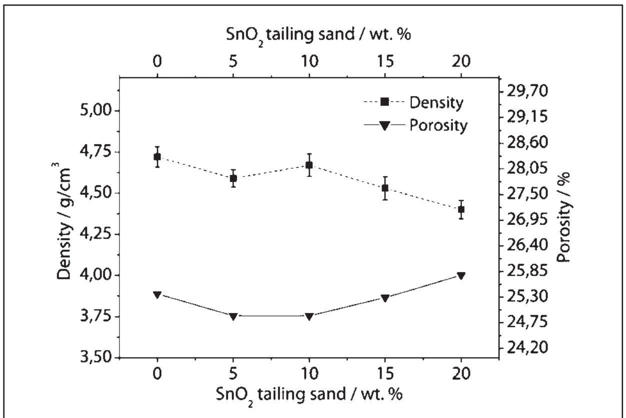

Figure 3 Variation of density and porosity composites after sintering as a function of the SnO_2 tailing sand content.

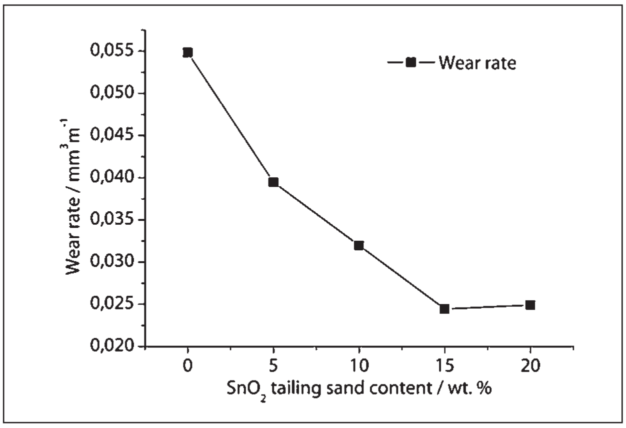

Figure 5 The effect of the SnO_2 tailing sand content on the wear rate of composites after sintering.

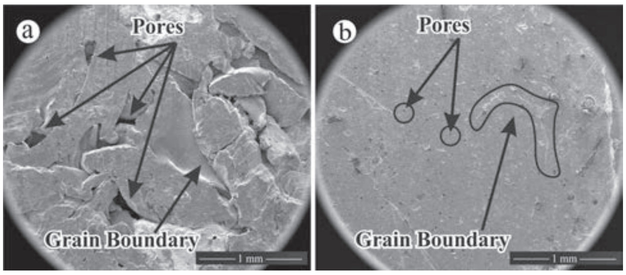

Figure 6 SEM image of unreinforced Cu10Sn morphology (a) before sintering and (b) after sintering.

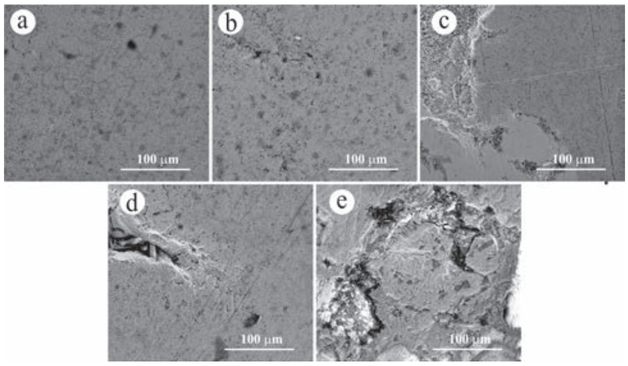

Figure 7 SEM micrograph of specimens sintered.

The SEM image of the SnO_2 tailing sand/Cu10Sn composites sintered is presented in Figure 7(a-e). The microstructure of 20 % SnO_2 tailing sand, as shown in Figure 7(e) indicates that the reinforcing is not homogeneous and agglomerate. The more volume fraction of SnO_2 tailing sand added, the more porous the composite was. However, this investigation supports of the porosity test value, as the one shown in Figure 3.

CONCLUSIONS

SnO_2 tailing sand which is dominated by $ZrSiO_4$ and SnO_2 has been used to strengthen Cu10Sn composites. The composites are produced by using warm compaction with multiple press methods. The results confirm that the density slightly decreased with the increasing content of SnO_2 tailing sand in the composites due to the presence of a low-density particle. It corresponds with the porosity of SnO_2 tailing sand/Cu10Sn composites which marginally increased compared to unreinforced Cu10Sn. Other than that, the hardness value of Cu10Sn composites increased with the increasing content of SnO_2 tailing sand. However, the hardness of composite dramatically decreased with 20 weight % reinforcement in Cu10Sn matrix.

Qualitative investigation for metallographic showed inhomogeneous structure at 20 % SnO_2 tailing sand. The utilization of powder mixer apparatus greatly influenced the homogeneity of mixtures. Furthermore, the increasing SnO_2 tailing sand amount resulted in a decrease in weight loss of composites. Eventually, SnO_2 tailing sand can enhance the wear resistance of Cu10Sn.

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Note: The professional translator for English is Ardian Setiawan, Malang, Indonesia