THE INFLUENCES OF POWDER MIXING PROCESS ON THE QUALITY OF W-CU COMPOSITES

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ABSTRACT

The mixing homogeneity of powders has a significant influence on the quality of composites fabricated from a powder metallurgy process. The factors that influence the homogeneity include the powder mixer, the medium, and the ball-milling procedure. In this paper, the influences of powder mixers and mixing media on the quality of tungsten-particle reinforced copper matrix composites are studied. Apart from dry mixing, other media used in the wet mixing process include n-butyl alcohol, camphor oil, and paraffin oil. Comparisons on mixing feature are made to a TURBULA® mixer and a Random mixer. The TURBULA® mixer is commercially available, and the Random mixer is an in-house designed machine. Our results show that using paraffin oil as the mixing medium, one may obtain optimal homogeneity in the composites. The Random mixer is superior to the TUBULA® mixer due to the fact that the Random mixer offers an avalanching motion creating pure shear forces onto the powders.

Keywords: Mixing homogeneity; powder mixer; ball milling.

INFLUCENCES DES PROCÉDÉS DE MÉLANGE DES POUDRES SUR LA QUALITÉ DES COMPOSITES W-CU

RÉSUMÉ

L'homogénéité du mélange des poudres influence de façon significative la qualité des composites produits par le procédé de métallurgie des poudres. Parmi les facteurs qui influencent l'homogénéité, il y a le mélangeur de poudre, le médium et le broyage par billes. Notre recherche porte sur les influences des mélangeurs de poudre et le médium sur la qualité des matériaux composites à matrice de cuivre renforcé de particules de tungstène. Mis à part le mélange à sec, les autres médium utilisés dans les procédés de mélange humide comprennent l'alcool de n-butyle, l'huile de camphre et l'huile de paraffine. Les comparaisons des caractéristiques des mélanges sont faites pour un mélangeur TURBULA® et un mélangeur Random. Le mélangeur TURBULA® est disponible en commerce, et le mélangeur Random est une conception maison. Nos résultats démontrent qu'en utilisant l'huile de paraffine comme médium de mélange, on peut obtenir une homogénéité optimale dans les composites. Le mélangeur Random est supérieur au mélangeur TUBULA® à cause du fait que le mélangeur Random offre un effet d'avalanche créant des forces de cisaillement pur sur les poudres.

Mots-clés : homogénéité du mélange; mélangeur de poudre; broyage par billes.

1. INTRODUCTION

Powder mixing as demonstrated previously [1–3] is an important step in the manufacturing process of many industrial products such as pharmaceuticals, foodstuffs, plastics, fertilizers, and ceramics. For years, tremendous work has been devoted to developing measuring techniques and models in particle technology. Modern technology of computerization, as pointed out in [4,5], has led to the development of phenomenological models based on particle dynamics simulations and to systemic models based on population balances which are now currently used at appropriate scales. Technical methods for measuring powder and mixing characteristics have also undergone development [6,7], some of these being used as on-line process measurements including particle size analysis. With the continuous improvement of these methods, the course is open for the development of coherent chemical engineering research in particle technology and, more specifically, in powder mixing [8]. However, despite this progress, there is still a discrepancy between the increasing research activity and the effective application of models and techniques with an engineering approach at the production process level.

Mixing can be described as a combination of three phenomena [9], namely: diffusion (viz. the motion of a particle with respect to its neighbors); convection (viz. the motion of a group of particles in relation to their neighbors); and shearing (viz. a change of distribution layers of ingredients in space). Blending is intended to ensure a uniform distribution of all components in the end product [10] In this way, each sample will contain an appropriate amount of the active components. There are many factors that influence mixing quality such as mixing time, speed of mixing rotation, the type of the mixer, dry or wet mixing process, and so on. It is acknowledged that mixing time and speed of rotation are to be controlled to ensure uniform mixing, improper mixing makes the ingredients distribute un-uniformly, and excessive mixing can work-harden the powders making the compaction difficult. This study aims at evaluating the effects of powder mixers and addition of mixing media on the quality of the powder composites.

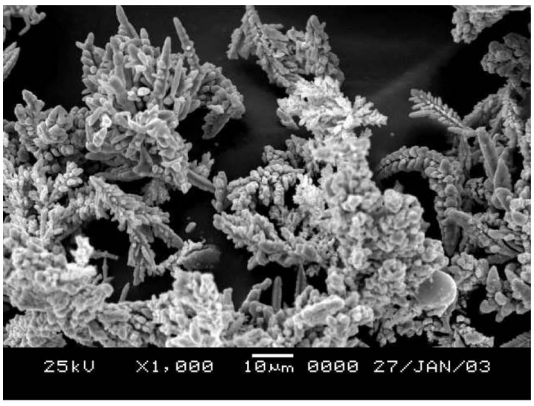
There are different methods to assess the performance of powder mixers. Traditionally, a volumetric sample thief with cavities on it is used to remove sample portions from different locations in the mixer. Unfortunately, several factors make the use of sampling thieves problematic [3]. One is that insertion of a probe disturbs the powder mixture, and another is that powders tend to segregate as they flow freely into the cavities. These two factors are known to make samples obtained by thief sampling different from the actual mixture composition [11,12]. Another limitation of the sample thief approach is that it is not practical to take more than 10–20 samples at a time, which are too few to accurately characterize the state of a powder mixture, especially in cases of strong deviations from the normal distribution [13]. Therefore, methods to examine powder mixtures in situ, non-invasively are preferred.

In the present study, the performance of two powder mixers is compared. The difference between the two mixers is their kinematical mechanisms in mixing the powders; one offers drop-impact loads and the other avalanching effects. The powders considered herein are tungsten-particle reinforced copper matrix composites (W-Cu composites) of which the relative densities and electrical conductivities are considered as the criteria for comparison. Vacuum oil-dripping method and four-point probe technique are employed to evaluate the density and the electrical conductivity, respectively. The powder images are acquired with optical and scanning microscopy. During powder mixing, n-butyl alcohol, camphor oil, and paraffin oil are used as mixing media. The influence of these media is also studied.

2. EXPERIMENTAL PROCEDURE

2.1 Composite preparation

The W-Cu composite powders fabricated in this study comprised dendrite electrolytic Cu powder (99.5% purity, 20 μm particle size) mixed with W particles with an average particle size of either 1 μm or 20 μm. The SEM images of the dendrite electrolytic raw Cu particles, the



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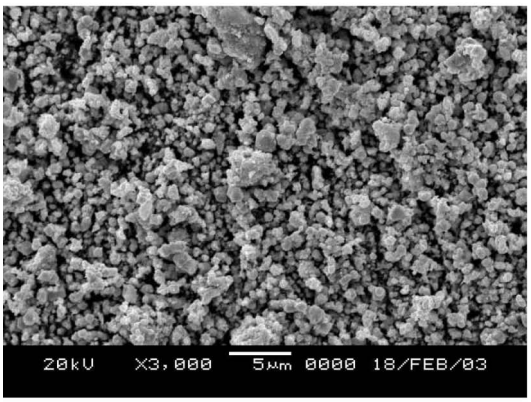

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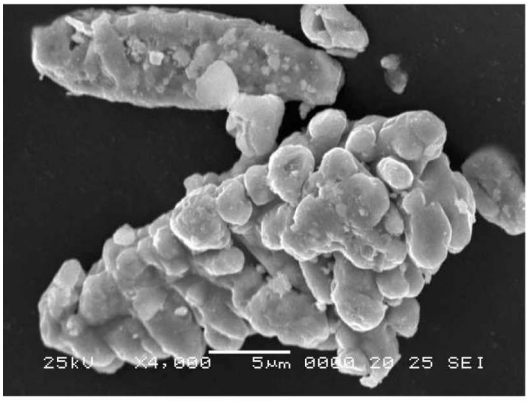


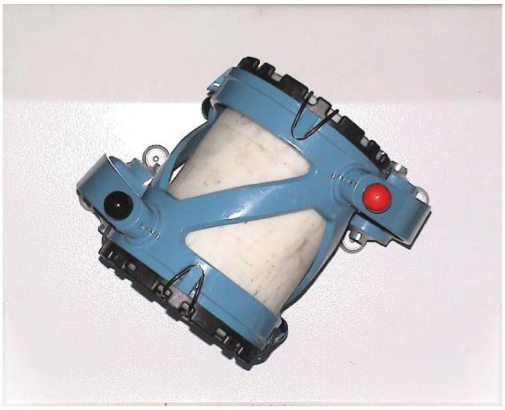
(c)

Fig. 1. SEM images showing morphologies of: (a) 20 μm Cu particles, (b) 1 μm W particles, and (c) 20 μm W particles.

1 μm raw W particles, and the 20 μm W particles are shown in Figure 1(a), 1(b), and 1(c), respectively. The W powders and Cu powders are mixed through a dry ball-milling process and a wet ball-milling process, respectively.

During the mixing process, ZrO_2 - Y_2O_3 (TZP) balls of sizes of 1mm, 2mm, and 5mm, with ball weight ratio 7:2:1, respectively, are used. The balls are poured into the shaker to half volume of the shaker before the powders (about 500 grams) are filled in. The mixing process lasts for 24 hours. In the dry ball-milling process there is no mixing medium added. The powders are mixed in air without lubrication and cooling. On the other hand, three media are used in the wet ball-milling processes; they are n-butyl alcohol, camphor oil, and paraffin oil. These media provide lubrication and cooling. In addition, their polarization disperses the agglomerates due to static electrical forces.

Two kinds of mixers are used for mixing in this study. The TURBULA® mixer shown in Fig. 2(a) is manufactured by Willy A. Bachofen (WAB) Company. In this mixer, the powders are mixed by rotation, translation, and inversion according to the *Schatz geometric theory* [14]. Although the mixer is claimed to be high efficient and can mix the powders quickly, it offers



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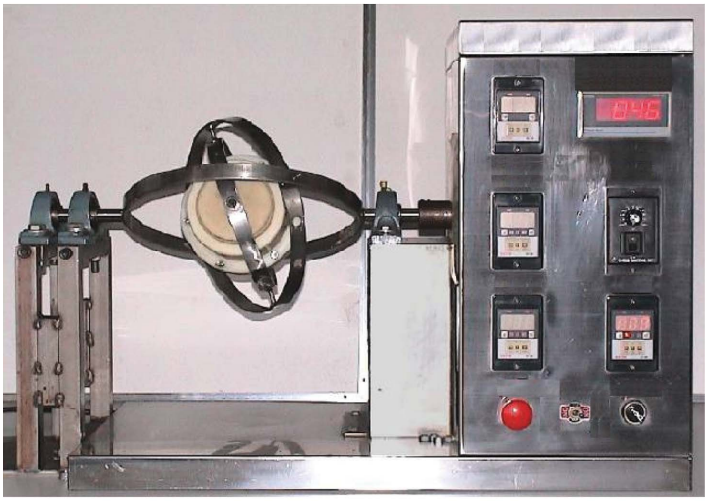


(b)

Fig. 2. The powder mixer ; (a) the TURBULA® mixer and (b) the Random mixer.

continuously drop-and-impact effects through the milling balls onto the powders. Consequently, the composite particles are deformed in flake forms and large thin chunks as a result of cold-weld. The second mixer, called the Random mixer, is an in-house designed mixer as shown in Fig. 2(b). The mixer moves within rotational frames which can rotate in three perpendicular directions. Its spherical shaker is able to rotate smoothly along three axial directions. Consequently, the milling balls and the powders in the shaker are moving as avalanche in which pure shearing and friction effects are present. This avalanche motion is believed to be able to disperse the agglomerates thus improve mixing homogeneity as the motion creates mainly shear but little impact effects. This feature of avalanche motion helps the powders keep their original shapes.

The processing, including schedule, is shown in Fig. 3. The composite powders are molded at a pressure of 450 Mpa before being pre-sintered at 900°C for one hour. The powders are then sintered at 1050°C for one hour. Next, a re-pressing process at 750 Mpa follows. Finally, the composite powders are re-sintered at 1050°C for one hour. The powder morphology, the mechanical and electrical characteristics of the composite are of interest.

2.2 Density measurement

The densities of the re-pressed/re-sintered W-Cu samples were measured using a vacuum oil-dipping method based on Archimedes principle. The theoretical density of the composite powder is given by

$$d_t = d_1 \cdot V_1 + d_2 \cdot V_2 = \frac{d_1 \cdot d_2}{d_1 w_1 + d_2 w_2} \tag{1}$$

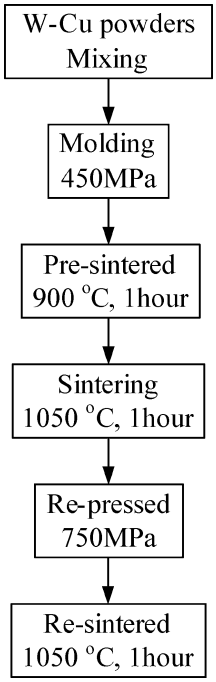


Fig. 3. Process schedule, including sintering, for W-Cu composite samples.

where d_1 and d_2, V_1 and V_2, and w_1 and w_2 are the theoretical densities, the volume fractions, and the weight fractions of the two phases. The experimental density of the composite powder, denoted as d_e, is given by

$$d_e = \frac{w_{\text{sample}}}{(w_{\text{oil}} - w_{\text{water}})} d_t, \tag{2}$$

where w_{sample}, w_{oil}, and w_{water} are the weights of the sample, of the sample with oil and of the sample with oil in water, respectively. Note that the density is in g/cm^3, the volume is in cm^3, and the weight is in gram throughout the study.

2.3 Electrical conductivity measurement

The electrical conductivities of the composite powders were determined using a four-point probe method. The resistance, R (measured in ohm), in the gauge length of each sample was measured using a micro-ohm gauge. The specific resistance, ρ, was then determined in accordance with

$$\rho = \frac{RA}{L}, \tag{3}$$

where A (in cm^2) is the cross-sectional area of the sample and L (in cm) is the gauge length. The electrical conductivity, E_c, was then computed as

$$E_c = 1/\rho, \tag{4}$$

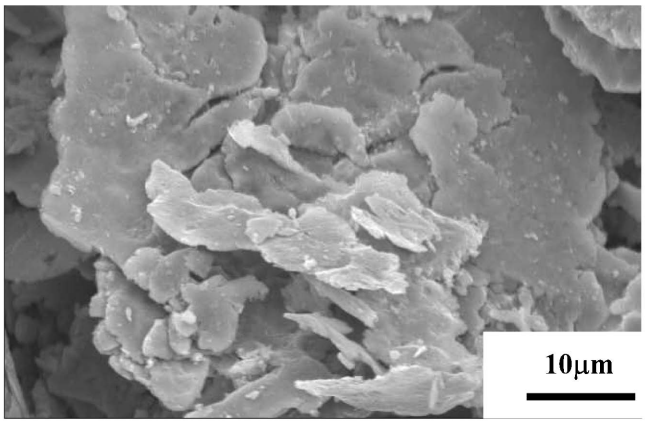
Finally, the electrical conductivity was converted to the International Annealing Copper Standard (IACS), i.e. %IACS = E_c / (5.8×10^7 $\Omega^{-1}\text{m}^{-1}$).

3. RESULTS AND DISCUSSION

3.1 The influences of the mixers

Fig. 4(a) shows the image of the W-Cu powders fabricated with the TURBULA® mixer and Fig. 4(b) with the Random mixers. The W particles are 20 μm. The paraffin oil is used as the medium and the powders are ball-milled for 24 hours resulting in a refined microstructure [15,16]. The powder shown in Fig 4(a) is in a flake shape with significant irregularity. On the other hand, the powder in Fig. 4(b) has dendritic shapes of Cu powders. The flake shape with irregularity suggests that the powders are subjected to drop and impact effects offered by the TURBULA® mixer whose linkage mechanism delivers rotation, translation, and inversion. The drop-impact force transfers to the composites with a rapid and instantaneous strike which, in turn, deforms the copper powders in to a flake shape and introduces work hardening effects. Work hardening effect tends to deteriorate steel molds and to make it difficult to obtain an unbroken green compact as a result of the spring back during mold release. On the other hand, the dynamic mechanism of a Random mixer is the avalanching motion. This motion mainly creates shear forces. Therefore, powders are subject to pure mixing; in a sense, there is neither cold-welding nor deformation. As a result, the green compact is not damaged due to little spring back during mold release.

Fig. 5 presents the SEM image as well as the optical image of W-Cu composite powders after dry ball milling in the TURBULA® mixer for 24 hours. It can be seen in Fig. 5(a) that without added mixing medium the Cu powders are struck into flake shape and stacked into a layered



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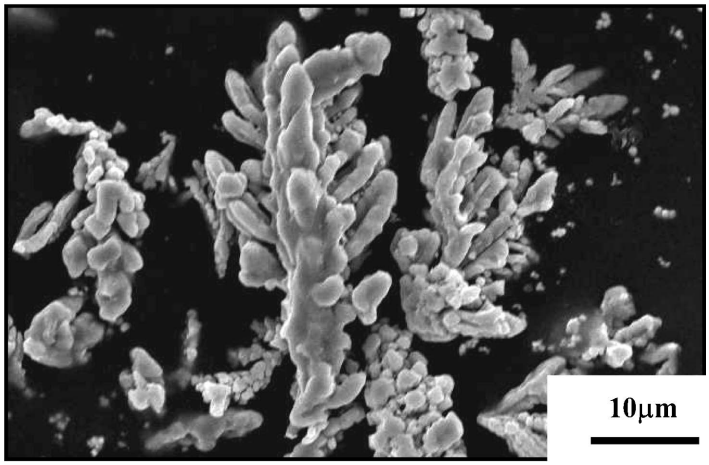

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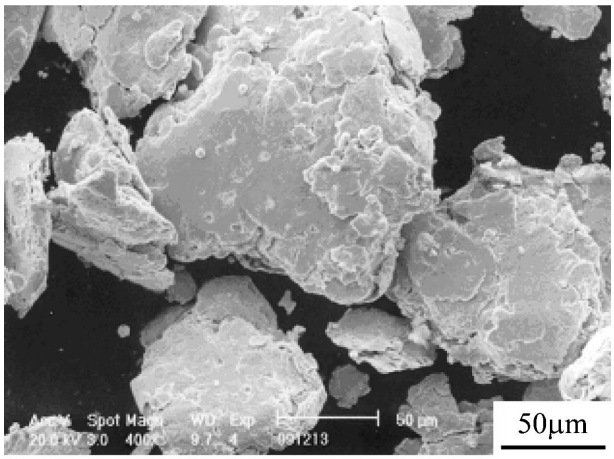
Fig. 4. SEM micrographs of W-Cu powder after ball milling for 24 hours with paraffin oil medium in (a) the TURBULA® mixer, and (b) the Random mixer. The W particles are 20 μm.

structure under a cyclic process of cold welding, deform, fragmentation, and cold welding. This observation has been described for a similar mechanical alloying mixing process [17]. During the process, the strengthening phase is embedded and cold-welded with the flake Cu powder. Although this kind of powders can be sintered into agglomerates with fine grains, there still exists poor sintering quality among the agglomerates. In addition, one of the major disadvantages is that the flake powders fabricated by the dry ball-milling process are readily cold-welded and left on the container wall, which make it difficult for an operator to scrape the composites off and to clean the container; although the composite could be scraped off, they are no longer usable. The powders seen in Fig 6 are fabricated through a dry ball milling process in the TURBULA® mixer for 24 hours. Fig. 6(a) shows the cold-welded composite powders left on the wall. Those composites that are not welded on the wall are in the form of loose chunks as shown in Fig. 6(b). Also notice that the pressed and released green compact shown in Fig. 6(c) has considerable cracks that make the green compact unusable. In addition, the compositions of the composites cannot be identified. In this study, the phenomenon is frequently observed in the dry ball- milling process with the TURBULA® mixer but occasionally in the random mixer. In



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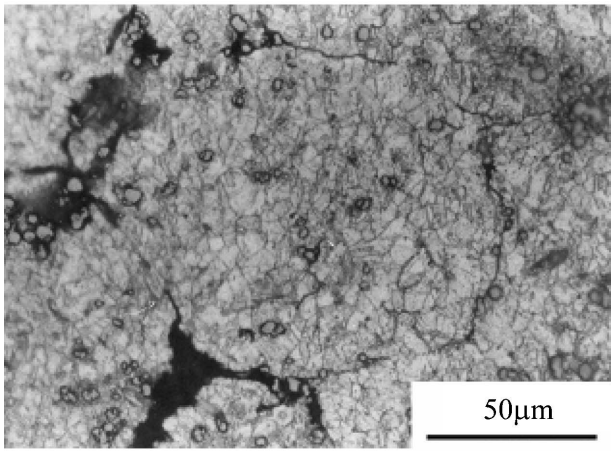

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Fig. 5. W-Cu powder after dry ball milling for 24 hours in the TURBULA® mixer. (a) The SEM image of powder morphology. (b) The optical image of the sintered microstructure.

light of the observations, the dry ball-milling process is not adopted in subsequent experiments of the study.

3.2 The effects of mixing media

The mixing homogeneity, σ, is determined based on the element composition detected by Energy Dispersive Spectrometer (EDS) in a prescribed area [18,19]. There are five areas considered. Each area is a square with side length of 500 μm, 200 μm, 100 μm, 50 μm and 20 μm, respectively. Ten random detections are made on each square; thus, there are 50 detections. The mixing homogeneity is then taken as the average of these 50 detections.

Presented in Figures 7 and 8 are the results showing the effects of the media on mixing homogeneity for the composites either with 1 μm or 20 μm W particles; each composite comprises 3 vol.% W addition. Fig. 7(a) presents the homogeneity results for 3 vol.% 1 μm W-Cu composite fabricated by the TURBULA® mixer and Fig. 7(b) the homogeneity by the Random mixer. Fig. 8(a) presents the homogeneity results for 3 vol.% 20 μm W-Cu composite fabricated by the TURBULA® mixer and Fig. 8(b) the homogeneity by the Random mixer. As

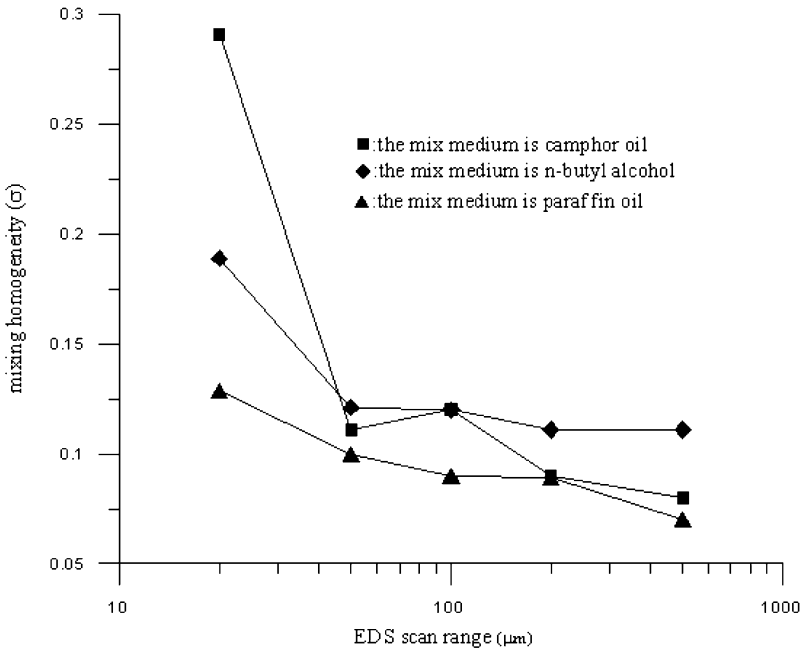
(a)

(b)

(c)

Fig. 6. The photographs of W-Cu powder after dry ball milling for 24 hours. (a) Cold-welded powders left on the container wall. (b) Residual powders in large loose chunks. (c) Cold-pressed and released green compact with considerable cracks.

described previously [19], a high value of σ (σ: mixing homogeneity) indicates a poor homogeneity. It is easily seen from Figs. 7 and 8 that as the EDS scan range gets larger, the homogeneity tends to be smaller and closer regardless whatever mixer and medium are used. On the other hand, as the EDS scan range gets smaller, especially for 20 μm W-Cu composite (Figs. 8(a) and 8(b)), the homogeneity deviates significantly for all media. This is because as the scanned area gets small, say 20 μm × 20 μm, the area is almost the same as the entire scope of



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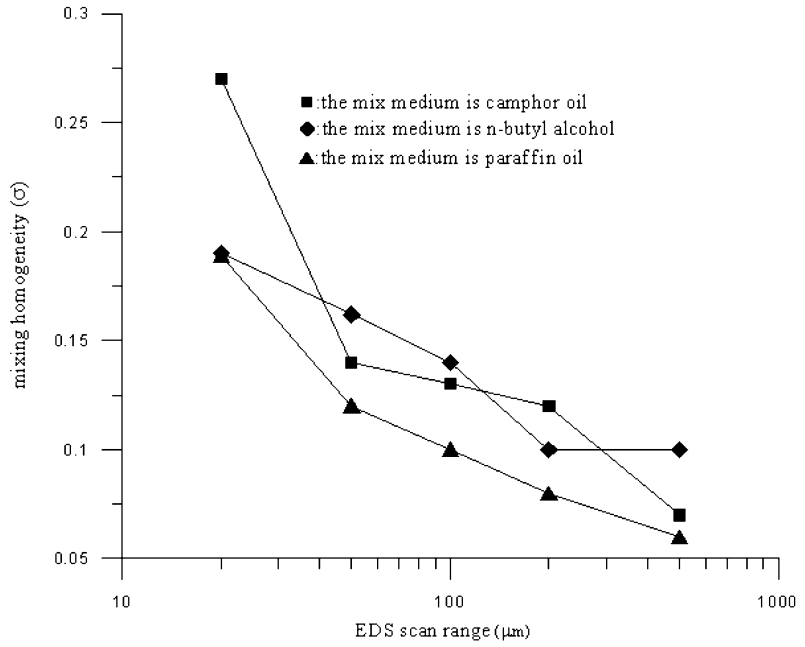

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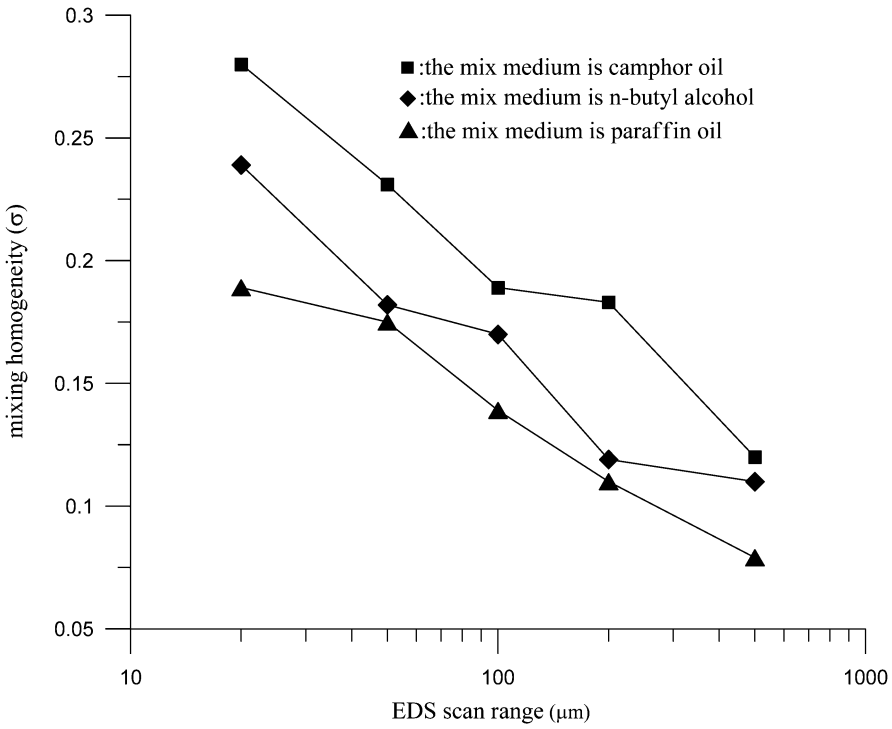
Fig. 7. The influence of mixing media on the mixing homogeneity for 3 vol.% 1 μm W-Cu powders. (a) Powders fabricated in the TURBULA® mixer. (b) Powders fabricated in the Random mixer.

a raw copper particle. Thus, there is an extremely high probability that only the copper base or the strengthening phase is scanned.

The media used in the mixing process include n-butyl alcohol, camphor oil, and paraffin oil. Again, one can see from both Figs. 7 and 8 that there is a lowest σ value when paraffin oil is used as the mixing medium. The fact that paraffin oil has a higher viscosity might be responsible



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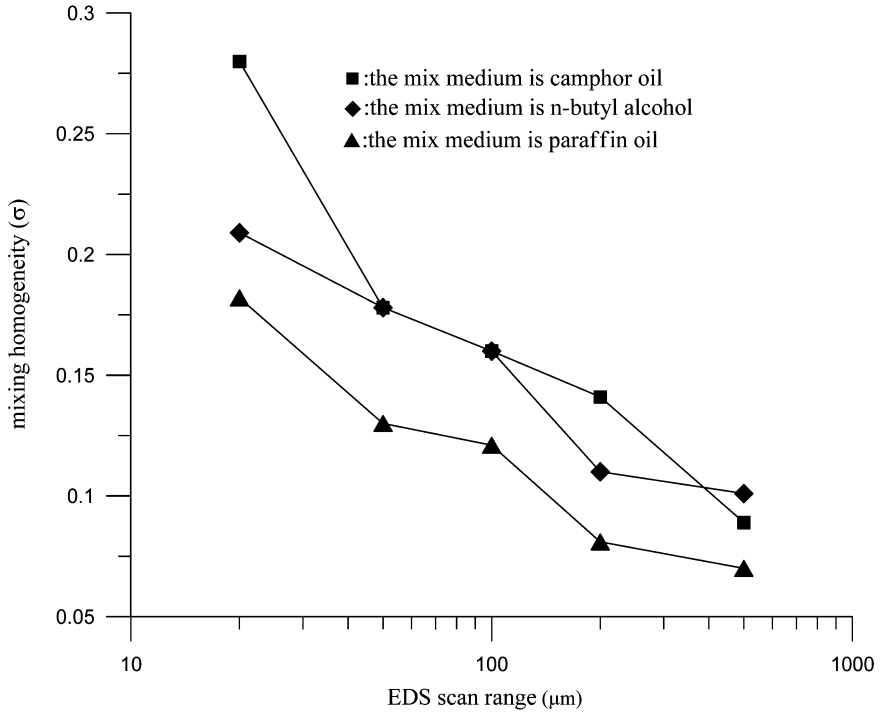

(b)

Fig. 8. The influence of mixing media on the mixing homogeneity for 3 vol.% 20 μm W-Cu powders. (a) Powders fabricated in the TURBULA® mixer. (b) Powders fabricated in the Random mixer.

Table 1. The fractional density of the 3vol.% W-Cu composite with different mixers and media.

W-particle size	process	Fractional density (%)					
		NR	PR	CR	NT	PT	CT
1 μm	sintering	93.85	94.82	88.35	89.75	88.78	89.21
	Re-pressed/re-sintered	97.30	98.06	95.25	95.14	96.44	95.68
20 μm	sintering	87.48	92.45	86.84	88.13	84.48	88.99
	Re-pressed/re-sintered	93.61	93.85	91.08	93.56	94.06	92.45

Notations: N: n-butyl alcohol, P: paraffin oil, C: camphor oil, R: in the Random mixer, T: in the TURBULA® mixer.

for this outcome. During the mixing process, when a hardened particle is separated from an agglomerate, it is difficult for the particle to return to the original or become a part of a new agglomerate if the medium has a high viscosity. On the other hand, although both n-butyl alcohol and camphor oil have polarity, due to their low viscosity the separated particles are likely to compact with other particles.

3.3. Results of the fractional density

In this study, the fractional density of the composite is of interest to characterize W-Cu composites. Table 1 shows the fractional density of the 3 vol.% W-Cu composite with different mixers and media. The results indicate that the composite fabricated under wet ball-milling in the Random mixer with paraffin oil as the medium has the highest fractional density. This result also reveals a similar trend on homogeneity discussed in Sec. 3.2; That is, a lowest σ value can be obtained when paraffin oil is used as the mixing medium. Hence, it suggests that there is a close relationship between the homogeneity and the fractional density. Table 1 also shows that the overall fractional density of the composite with 1 μm tungsten particles is higher than that of the composite with 20 μm tungsten particles.

3.4. Results of the electrical conductivity

In Table 2, the influences of the mixing process on the electrical conductivity of the 3 vol.% W-Cu composite are presented. Based on the values of electrical conductivity in Table 2, it is

Table 2. The conductivity of the 3 vol.% W-Cu composite with different mixers and media.

Mixer	W-particle size	Conductivity (%IACS)		
		NBU[a]	PAR[b]	CAM[c]
Random	1 μm	86	85.8	84.5
	20 μm	84.2	88.9	86.2
TURBULA®	1 μm	85.9	83.5	82.5
	20 μm	81	85.2	84

a: the mix medium is n-butyl alcohol
b: the mix medium is paraffin oil
c: the mix medium is camphor oil

considered that the composite fabricated in the Random mixer with paraffin oil as the medium has a better electrical conduction. Notice that the overall conductivity is lower than 90 %IACS. This is because there exists poor bonding between the base and the strengthened particles which lack of electroless copper plating process [20]. Interestingly, it is noticed from Table 2 that when using the paraffin oil or camphor oil as the medium, the composite with 20 μm strengthening particles possesses a higher electrical conductivity than the composite with 1 μm strengthening particle. This phenomenon is realized from a view point of the total area of strengthening phase/base as larger strengthening particles tend to reduce the total area. However, one may also notice that the composite with 1 μm strengthening particles with the n-butyl alcohol as the medium possesses a higher electrical conductivity.

4. CONCLUSION

In this paper, the influence of powder mixers and mixing media on the qualities of W-particle reinforced Cu-matrix composites is studied. From the results it may conclude that the Random mixer is superior to the TURBULA® mixer. The Random mixer offers an avalanching motion that mainly creates shear forces such that the composites are subject to mixing only without either cold-welded or deformation. In addition, the two mixers predict similar homogeneity results when the scanning area is larger (e.g. 200 or 500 μm side length). However, as the scanning area gets smaller (e.g. 50 or 100 μm side length), the Random mixer provides a higher homogeneity value. One disadvantage of using the TURBULA® mixer is that work hardening is introduced on the copper powders. The results also suggest that the wet ball-milling process is better than the dry ball-milling process. An optimal homogeneity of the composite is achieved through a wet ball-milling process with paraffin oil as the medium because paraffin oil possesses a higher viscosity. The composite fabricated under wet ball-milling process with paraffin oil as the medium has the highest fractional density, which holds a close relationship with powder homogeneity. Finally, the composite fabricated in the Random mixer with paraffin oil as the medium is consider better in terms of electrical conduction. Moreover, it may conclude that composites with 20 μm strengthening particles possess a higher electrical conductivity when either paraffin oil or camphor oil is used as the medium.

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APPENDIX

List of notation

d_1, d_2:	Theoretical densities of the two phases in composite powder
d_e:	Experimental density of the composite powder
d_t:	Theoretical density of the composite powder
w_{sample}:	Weight of the sample
w_{oil}:	Weight of the sample with oil
w_{water}:	Weight of the sample with oil in water
A:	The cross-sectional area of the sample
E_c:	Electrical conductivity
L:	Gauge length
R:	Resistance in the gauge length

V_1, V_2:	Volume fractions of the two phases in composite powder
%IACS:	Percent electrical conductivity in International Annealing Copper Standard
ρ:	Specific resistance
σ:	Mixing homogeneity