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Research Article

Influence of Polyethylene Glycol and Methanol Additions on the Properties of Ball-Milled Cu4B4C Composite Powders

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Abstract:

This study investigated the effect of different process control agent (PCA) usage on mechanical alloying behavior of boron carbide (B_4C) reinforced copper (Cu) based composite powder. For this purpose, elemental Cu and B_4C powders were weighed and powder specimens were prepared with respect to appropriate mass ratios (96% Cu and 4% B_4C). Two different PCA additives, namely polyethylene glycol (PEG) and methanol, were also used to prepare powder samples. The amount of PCA was kept constant at 5wt.% for both specimens. These prepared powder samples were then milled using a planetary type ball-mill. After specified milling periods, milling runs were interrupted and powder samples were extracted from the milling vials for further powder characterization including powder morphology using scanning electron microscopy (SEM) and average particle size (APS) via laser diffraction analysis (Mastersizer). Accordingly, after the completion of milling runs, i.e., 15 hours of ball-milling, methanol addition was found much more effective than PEG at reducing particle size. Final APS values for powder specimens having PEG and methanol as PCAs were determined to be 8.237 and 4.101 μm, respectively.

1. Introduction

Composite materials are used in a variety of applications, such as machinery, automotive, railway, marine, aviation, defense, ballistics, construction, biomedical, electrical and electronics due to their outstanding properties compared to those of conventional materials [1-8]. They compose mainly of matrix and reinforcement phases, and they possess distinct properties depending on the combination of the constituent elements. Accordingly, they may be classified as metal-matrix composites (MMCs), ceramic-matrix composites (CMCs), and polymer-matrix composites (PMCs) [9-15].

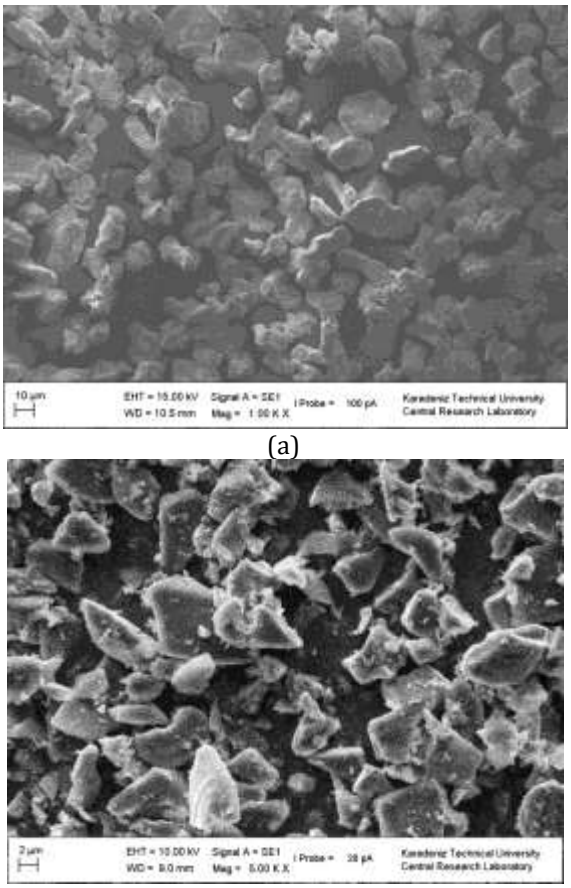
Powder metallurgy (PM) technique is frequently used to produce MMCs. Production method plays an important role on material properties. Namely, identical materials can have very different properties due to the differences in their manufacturing technology. Apart from this, a change in production route or operation sequence affects material properties as well. For example, powder processing route affects homogeneity, size and shape of powder mixtures. Therefore, mechanical alloying (MA) technique is emerged as an effective way to prepare powder mixtures or even to manufacture nanocomposite powders [16-19]. However, MA method has several process parameters to be optimized to enhance process efficiency [20-21]. Otherwise, it will take much longer times and higher production expenses to fabricate a desired material. Therefore, MA parameters must be optimized for a specific composition. Among these, type and amount of process control agent (PCA) is one of the most important parameter to provide size reduction of powder particles [22-25]. PCAs used for MA experiments may be solid (stearic acid, polyethylene glycol (PEG), and zinc stearate) or liquid (ethanol and methanol) type. Other parameters include milling speed, ball-to-powder weight ratio (BPR), diameter and material of grinding balls, vial capacity and material, type of mill, pause times, reverse mode and milling duration.

In this study, copper (Cu) metal is used as matrix material owing to its high electrical and thermal

conductivities, high formability and low cost making it one of the most important base material along with silver (Ag) to produce electrical contact materials [26-30]. However, to further improve its physical and mechanical properties some metal oxides, carbides or refractory metals are used as reinforcing materials [31-43]. For this aim, boron carbide (B_4C) powder is used to reinforce Cu matrix. Additionally, to observe morphological evolution and size reduction of ball milled powders; two different PCAs, namely PEG and methanol, were separately utilized for each powder specimen. Thereby, the effect of these PCAs on particle size and morphology was comparatively studied, and the most effective type of PCA was determined.

2. Material and Methods

In this study, elemental Cu (max. 44 μm, 99.0%) and B_4C (max. 10 μm, 99.7%) powders were used to prepare powder mixtures. Morphology of as-received powders was investigated by means of scanning electron microscopy (SEM) on a Zeiss Evo LS 10 model, and corresponding SEM images were presented in Fig. 1. Both powders were supplied by Alfa Aesar Corporation. The characteristics of as-received or starting powders were given in Table 1. These powders were firstly weighed to a specified mass ratios (96:4). Afterwards, PEG and methanol agents were separately added in both specimens. The amount of PCA was kept constant at 5wt.% for both specimen. A two grinding station planetary-type ball mill (Fritsch Pulverisette 7) was used to carry out ball-milling processes. Accordingly, these two different powder specimens prepared were later placed into the tungsten carbide (WC) vials. Meanwhile, WC grinding balls having a diameter of 10 mm were also used as a grinding medium. Milling parameters were selected to be a milling speed of 300 rpm and a BPR of 10:1. Grinding mode was chosen to be 2 minutes of grinding and 1 minute of pause including a reverse mode for one milling cycle. The powders were ball milled up to 15 hours. Table 2 lists all milling parameters used to synthesize $Cu4B_4C$ composite powders. By the way, to control heat increments that occur through repeated cycles, milling runs were interrupted at least 30 minutes at specified time intervals (3, 6, 9, and 12 hours), and then two samples were taken from each vial to be used for further powder characterization including powder morphology using SEM and average particle size (APS) via laser diffraction analysis (Mastersizer). A laser diffractometer (Malvern Instruments Mastersizer 2000) was used to detect APS (d_{50}) values throughout the milling processes. Thereby, the evolution of shape and size of powders were comparatively determined for each specimen by SEM and Mastersizer, respectively. Moreover, energy-dispersive X-ray spectroscopy (EDS) was used to detect elemental distributions, and X-ray diffraction (XRD) was used for the primary characterization of phase composition.



(a)

(b)

Figure 1. *Initial morphology of a) Cu and b) B_4C powders.*

Table 1. *Type, size and purity of as-received powders.*

Type of powder	Max. particle size (μm)	Purity (%)
Cu	44	99.0%
B_4C	10	99.7%

Table 2. *Milling parameters used to synthesize $Cu4B_4C$ composite powders.*

Chemical composition	$Cu4B_4C$
Type and amount of PCA	PEG - 5 wt.% methanol - 5 wt.%
BPR	10:1
Milling speed	300 rpm
Grinding mode	2 minutes grinding, 1 minute pause cycles / reverse mode active
Diameter and material of the grinding balls	10 mm, WC
Type of ball-mill and vial capacity	Fritsch Pulverisette 7 / 80 ml vials
Milling duration	3, 6, 9, 12, and 15 hours

3. Results and Discussions

Considering SEM images shown in Figure 1, Cu powder particles have an irregularly shaped (Fig. 1a) whereas B_4C powder particles have an angularly shaped (Fig. 1b). Table 3 lists powder samples' APS values obtained by recording mainly after each specified milling time intervals. APS of Cu powder was determined to be 28.895 µm before milling processes. The curves showing the relationship between particle size and milling duration were plotted and shown in Figure 2. It can seen from Figure 2 that, no fluctuations in APS are observed through powder processing sequences. APS values tent to decrease with increasing milling duration. But the gap between the curves of these two specimen reaches its critical threshold value after a milling of 9 h.

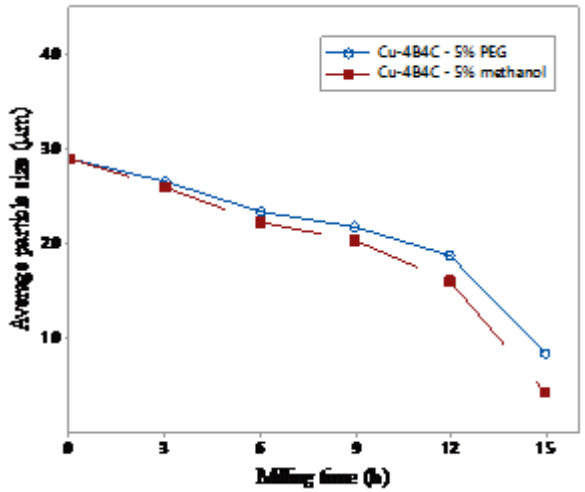


Figure 2. *APS variation of Cu4B$_4$C composite powders having PEG and methanol as PCAs*

Due to the differences in types of materials, they may be categorized as ductile or brittle. The utilization of PCAs is required to be used especially grinding ductile powders. On the other hand, in case of milling just brittle materials, the use of PCA may not be mandatory. The composition used in this study having both characteristics of powders, namely ductile Cu and brittle B_4C. Therefore, the usage of PCA will have positive effects on milling process and will shorten grinding times. Otherwise, without the use of PCA, severe cold welding among powder particles will counteract further decrease in powder particle size.

In the earlier stages of grinding, powder particles expose severe plastic deformation mainly exerted by ball-to-powder collisions. Therefore, the tendency of cold welding among ductile powders increases in the first 3 h of milling (Fig. 3a and b). With continued milling, the dominance of cold welded flaky particles lasts up to 6 h of milling (Figs. 3c and d). The trails of fracturing are apparently seen after milling duration of 9 h for the powder specimen having methanol (Fig. 3f). On the other hand, flaky particles are much higher in proportion with PEG usage (Fig. 3e) comparing to methanol after the same milling period. Therefore, there is a slight difference in APS values, i.e., 21.684 and 20.286 µm for the specimens milled with PEG and methanol agents, respectively.

Figure 4 shows SEM images reflecting the morphological evolution of powder mixtures after milling for 12 and 15 h. The effects of using different PCAs may be clearly seen at later stages of milling process. Fracturing effect continues to take place through the later stages of milling process. Accordingly APS values were reduced to 18.731 and 15.977 µm after a milling of 12 h. Comparing with PEG usage shown in Fig. 4a, the specimen milled with methanol exhibited more effective size reduction owing to the effectiveness in sustaining a balance between cold welding, shearing and fracturing (Fig. 4b). Effective use (type and amount) of PCAs is capable of synthesizing composite powders having different combinations of materials in relatively short processing times. This may be observed in the final morphologies of the powders (Fig. 4c and d). Accordingly, the differences in APS stem from methanol additive may be apparently seen from Fig. 4d.

Figure 5 shows the high magnification SEM images of the synthesized composite powders. Final APS values were quantified to be 8.237 and 4.101 µm for PEG and methanol usages, respectively. Corresponding SEM images prove that the composite powder produced by milling with methanol was exhibited much more uniform and fine distributed structure (Fig. 5b) than that the one in PEG (Fig. 5a).

Considering powder morphologies and APS values throughout the milling process, it was determined that the methanol addition significantly affected the final size and shape of powder particles. As a consequence, the use of methanol was found to be advantageous to reduce APS values as compared to PEG. This is important to produce high quality composites thanks to the improvement in microstructures, and therefore leading superior physical, mechanical, and thermal properties.

Moreover, EDS was used for mapping analysis (Fig. 6). In this map; red, green, yellow and turquoise represent the distribution of copper, boron, carbon, and oxygen elements, respectively. The weight percentages of aforementioned elements in the zone, shown in secondary electron (SE) image (Fig. 6a), were presented in Table 4.

Table 3. *APS values of $Cu4B_4C$ composite powders after each specified milling duration.*

Chemical composition	Milling duration (h) and average particle size (µm)					
	0	3	6	9	12	15
$Cu4B_4C$ - PEG	28.895	26.539	23.267	21.684	18.731	8.237
$Cu4B_4C$ - methanol	28.895	25.864	22.189	20.286	15.977	4.101

(PEG as PCA) **(Methanol as PCA)**

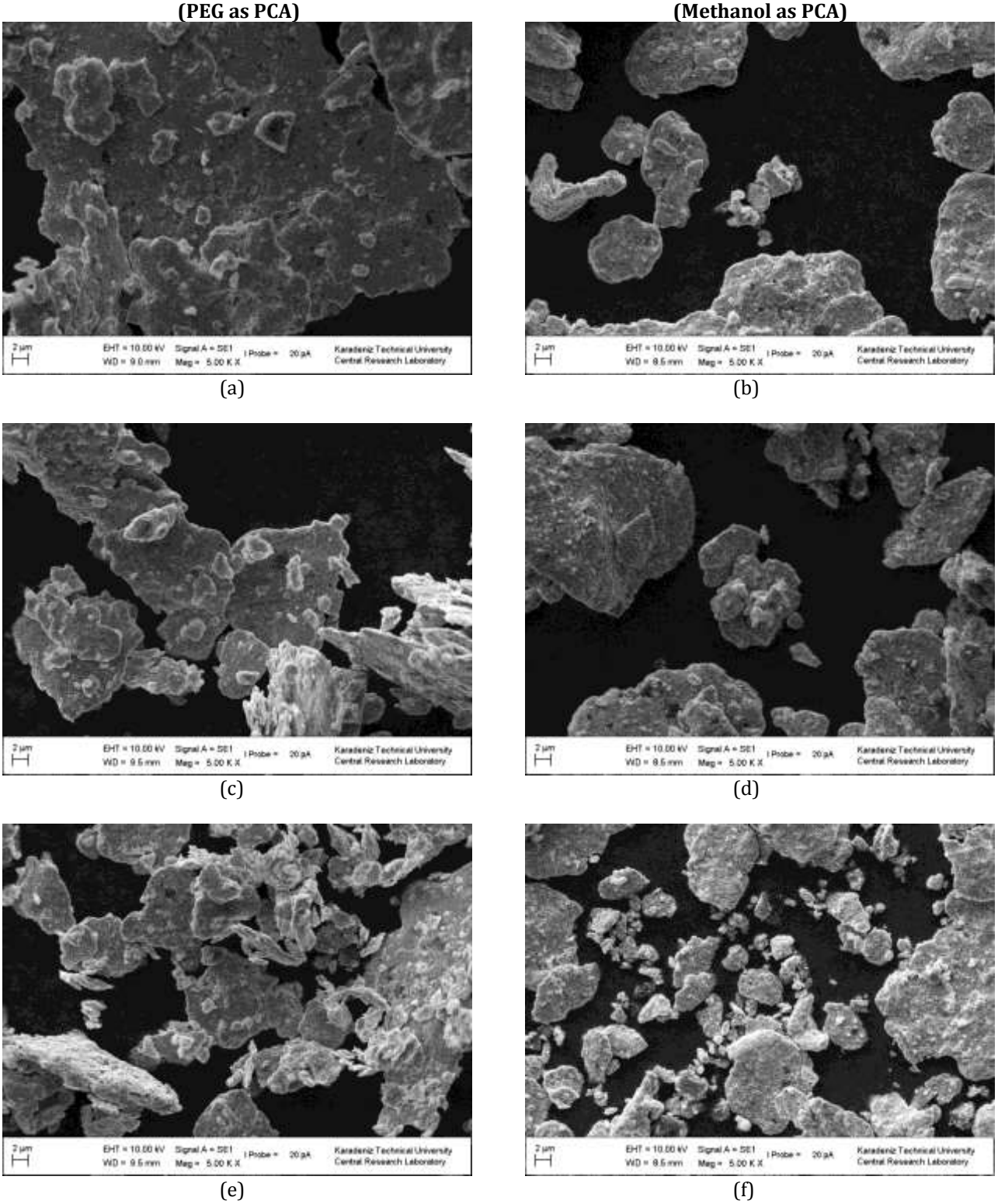


Figure 3. *SEM images showing morphology of $Cu4B_4C$ powder mixtures after milling for 3h (a-b), 6h (c-d), and 9h (e-f).*

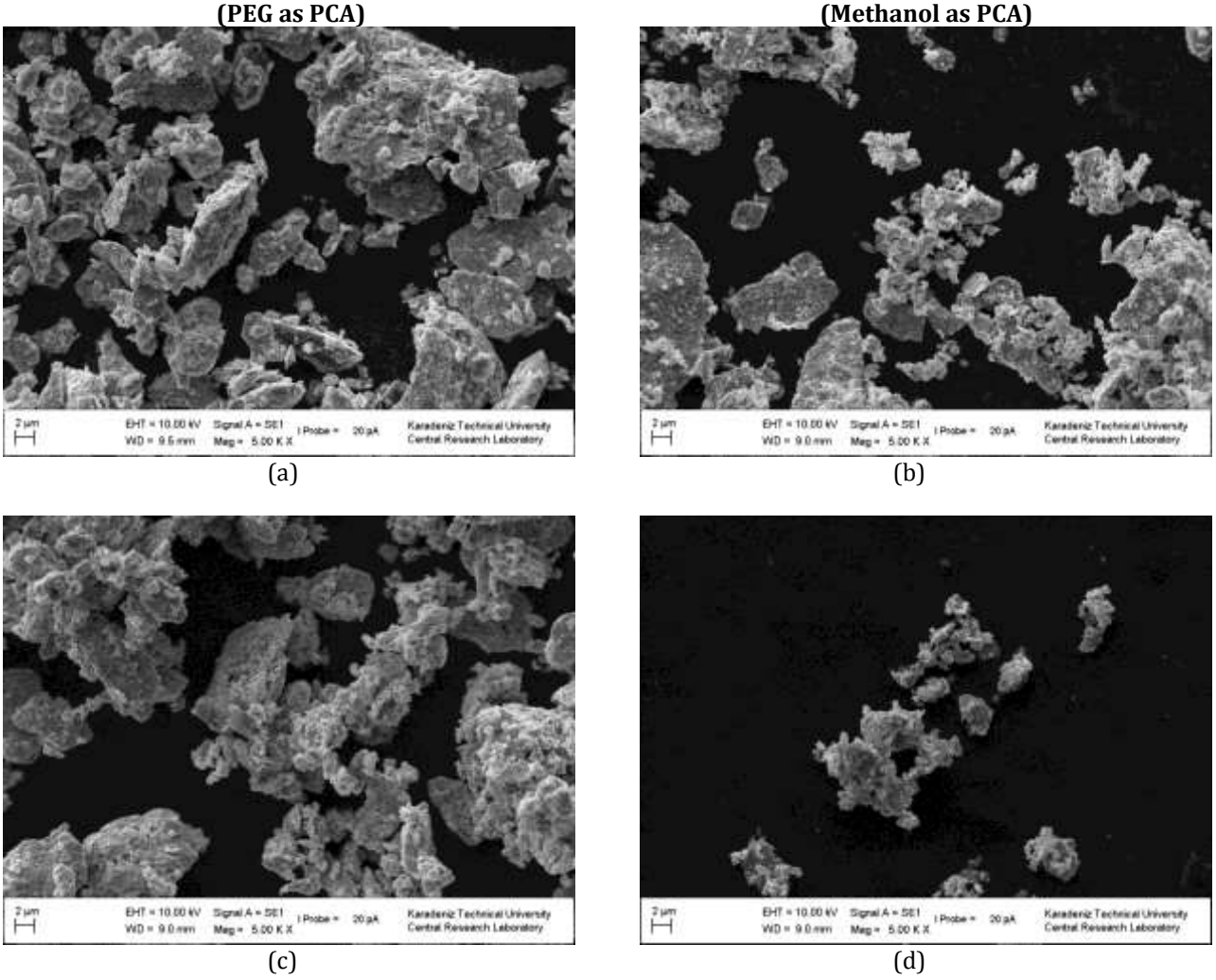


Figure 4. *Morphology of $Cu4B_4C$ powder mixtures after a milling for 12h (a-b), and 15h (c-d).*

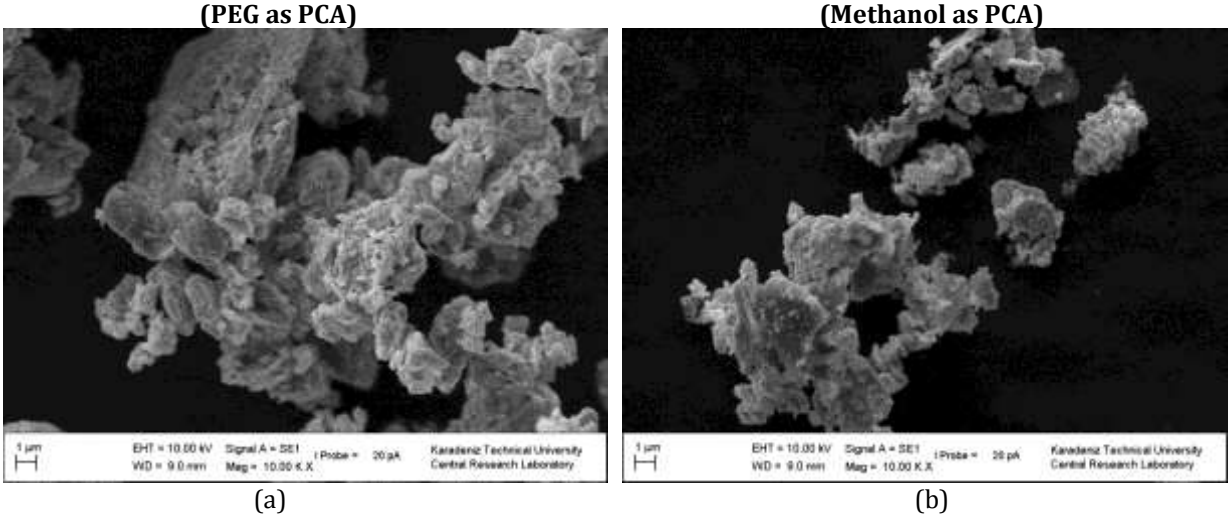


Figure 5. *High magnification SEM images representing final morphology of synthesized $Cu4B_4C$ composite powders.*

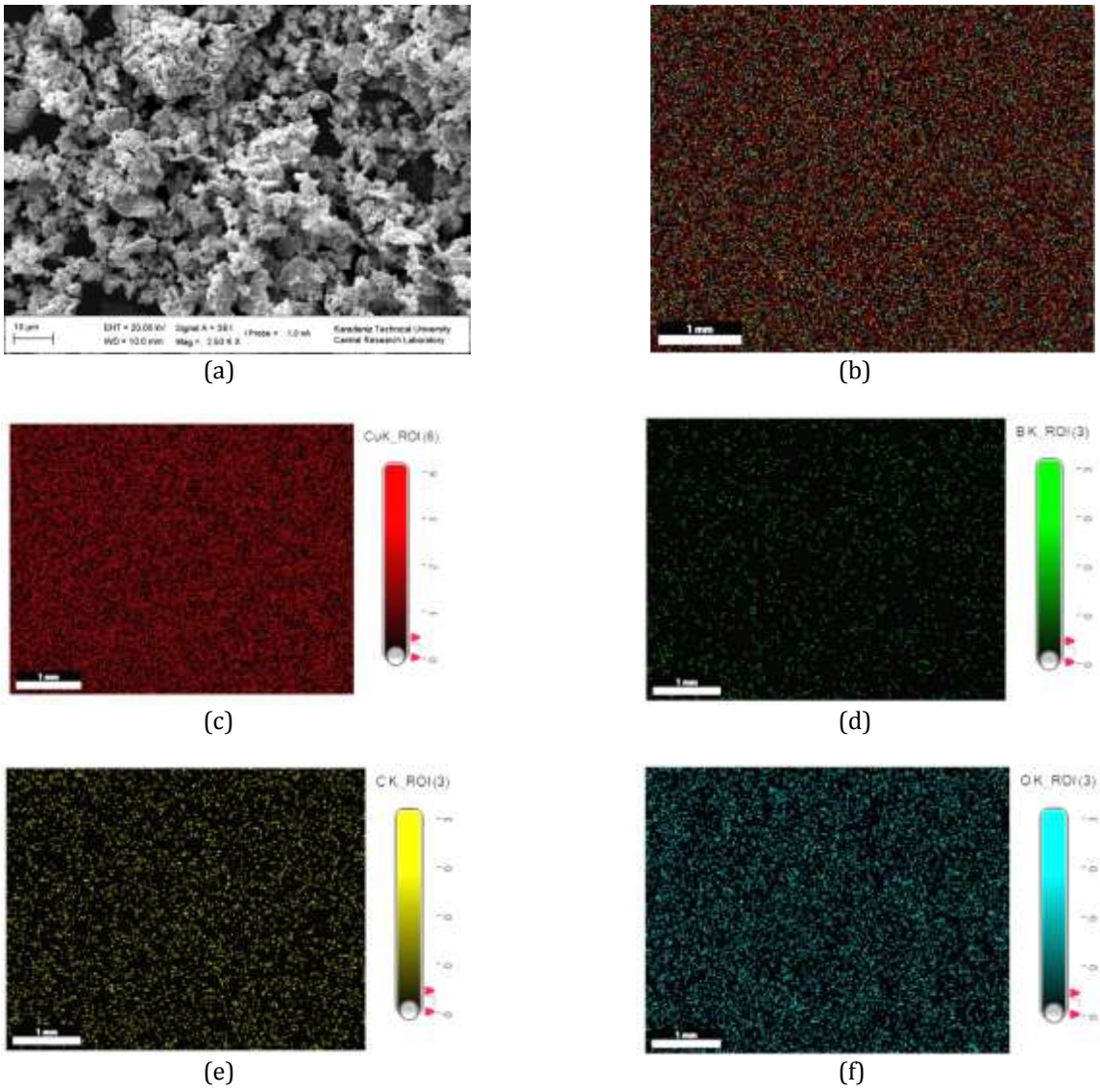


Figure 6. *EDS analysis of Cu4B$_4$C composite powder having methanol as PCA; secondary electrons (SE) image (a), distribution of the elements in the mapping zone (b), distribution of Cu element (c), distribution of B element (d), distribution of C element (e), distribution of O element (f).*

Table 4. *Chemical composition in EDS mapping zone.*

Element	Weight %
Cu	59.80
C	19.06
B	13.25
O	7.89

The weight percentages of Cu, C, B and O were recorded to be 59.80, 19.06, 13.25, and 7.89, respectively. As can be seen from Fig. 6, the homogenous dispersion of the elements is achieved after milling duration of 15 hours.

Figure 7 illustrates the XRD patterns obtained for Cu4B$_4$C composite powders. Here, black and red patterns represent the Cu4B$_4$C composite powders having PEG and methanol, respectively. Accordingly, Cu, B$_4$C and Cu$_2$O phases were formed. No peaks are detected for other impurity phases.

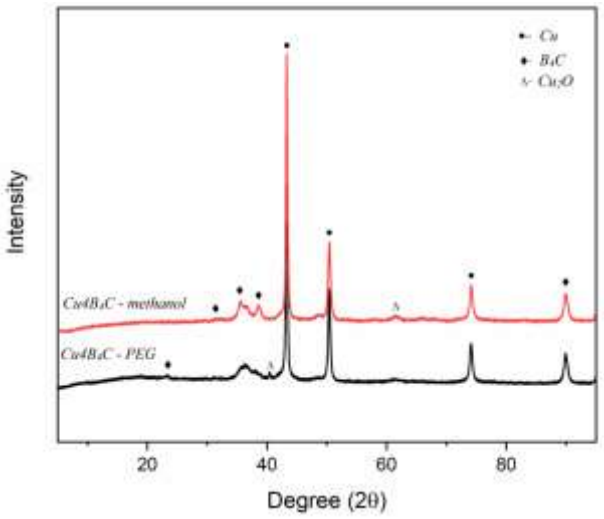


Figure 7. *XRD diagram of all composite powders.*

4. Conclusions

Copper (Cu) based boron carbide (B_4C) reinforced composite powders having two different process control agents (PCAs), namely polyethylene glycol (PEG) and methanol, were successfully synthesized by mechanical alloying (MA) technique. After the completion of milling runs, i.e., 15 hours of ball-milling, methanol addition was found much more effective than PEG at reducing particle sizes. Final APS values for powder specimens having PEG and methanol as PCAs were determined to be 8.237 and 4.101 μm, respectively. The powder specimen milled with methanol was exhibited much more uniform and fine distributed structure than that the one in PEG. This is important to manufacture high quality composites owing to the enhancement in microstructures, and thereby leading superior physical, mechanical and thermal properties.

Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The author declares that he has no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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- **Data availability statement:** The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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