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# Influence of mechanical alloying time on particle size of copper matrix composite

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## ABSTRACT

**Purpose:** The aim of this study was to determine the influence of mechanical alloying time on particle size of copper matrix composites. Particle size distribution is very important parameter in many research areas such as powder metallurgy, particle-based computational modelling, advanced nanocomposite materials, etc. Hence, knowledge of relations between particle size and applied technique is essential for many studies, especially for selection of further manufacturing procedures.

**Design/methodology/approach:** Starting powders (94.78 wt.% copper, 4.1 wt.% zirconium and 1.12 wt.% boron) were mechanically alloyed (MA) for 1, 10 and 20 hours. The structural characterization of copper and MA powders were performed by X-ray powder diffraction (XRPD) and morphology of MA powders were examined by using scanning electron microscopy (SEM). Particle size distribution as a function of milling time was determine by advanced laser nanoparticle sizer.

**Findings:** Obtained results show that with increasing milling time the particle size is decreasing and morphology is changing. Also, identification of nanoparticles was achieved. Analysis of particle size distribution point out that after 1 hour of mechanical alloying the particle diameter is decreasing until 10 hours after which it starts to increase.

**Research limitations/implications:** Identification of correlations between particle morphology/size distribution and milling time is of great importance in powder-based techniques and computational models.

**Originality/value:** Copper matrix composites reinforced with ceramic nano and micro particles are relatively new materials. Obtaining these kind of composite materials by powder metallurgy is new approach in its production. Optimization of mechanical alloying parameters for production of MA powders will provide control of final material properties.

**Keywords:** Composites; Mechanical alloying; Particle size distribution

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## MATERIALS

## 1. Introduction

Modern development of new materials requires not only determining their chemical composition, but also their structure, phase composition, physical and mechanical characteristics. It is also necessary knowledge of other characteristics, such as particles morphology, particle size distribution and specific surface area. Therefore, all these data concerning material properties leads to better understanding of material behaviour in different conditions, which is important for scientific as well as engineering standpoint [1-4].

Copper matrix composites have high values of electrical and thermal conductivity, as well as tensile and compressive strength which enables their wide use, mainly in electrical and automotive industry [4-9]. In recent years [9-12], much attention is devoted to the study of Cu matrix composites containing various alloying elements (zirconium, chromium, cobalt, titanium, boron, hafnium), in order to obtain suitable composite material for production of connectors, sockets, electrodes, etc. Powder metallurgy is technique which use powders as starting materials.

Hence, knowledge of particle size distribution is of great importance especially if is applying *in situ* technique for production composite materials with specific properties. Changing the powder properties may provide desirable final material properties [13]. Demanding conditions in industries need advanced materials with specific properties. In addition, production and implementation of these materials need to be cost-effective. Hence, characterization of particles and determining the correlations between particle sizes and milling time is fundamental for powder-based computational models and techniques. Since particle-based computational models can be effectively used for solving a variety of problems in engineering, particle size measurements are of great interest in many studies. Laser diffraction method conducts reliable and repeatable results, accordingly it is very often primary method for investigation of particle size distribution [14,15].

In this study an attempt was made to determine influence of mechanical alloying time on particle size of copper matrix composite. Advanced laser nanoparticle sizer with an inverse Fourier optic was applied for measuring particle size distribution.

## 2. Experimental procedure

Copper powder with the addition of 4.1 wt.% zirconium and 1.12 wt.% boron was homogenized for 1 hour in Turbula Type 2TC Mixer with stirring speed of 500 rpm. Homogenized mixture was mechanically alloyed in Netzsch attritor mill (Fig. 1) under an inert atmosphere (argon) for 1, 10 and 20 hours with steel balls 6 mm in diameter, at stirring speed of 330 rpm. Ball-to-powder weight ratio was 5:1. Changing the size of the powder particles applying mechanical alloying in attritor depends on a number of factors, such as [16]: the impact force of ball to powder, the rotation speed of blades, the mechanical properties of elemental powders, the amount of powder, the content of alloying elements, duration of the process, etc. The starting mixture for mechanical alloying must contain at least one ductile metal powder (copper in this case) which would serve as matrix. Also, it is important that one of the particles of the starting materials can be deformed and fractured when they are between two balls (Fig. 2) and due to high pressures in collision. Mechanical alloying was carried out in order to obtain mechanical activation of Zr and B particles and their proper distribution in the copper matrix.

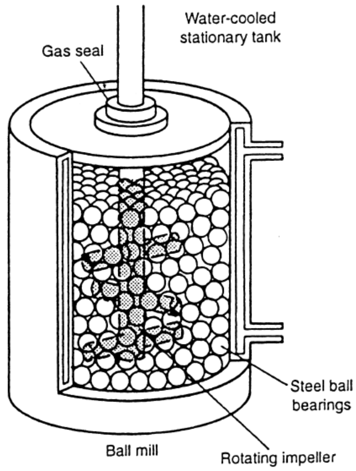


Fig. 1. Attritor ball mill [16]

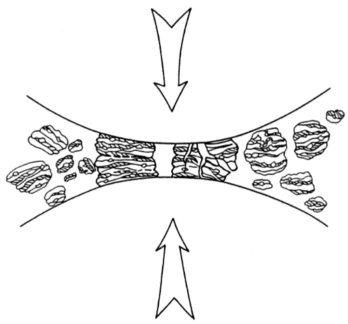

Fig. 2. The collision ball – powder particles – ball during mechanical alloying [16]

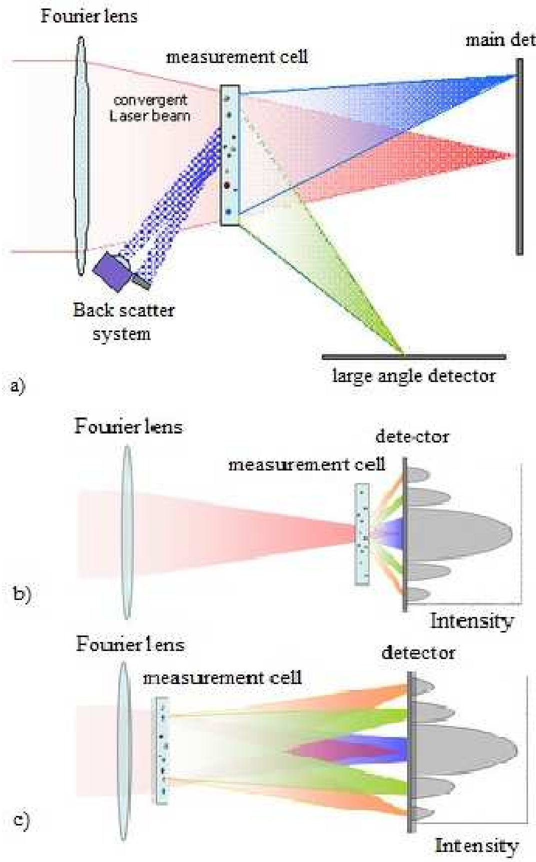


Fig. 3. a) The basic outline of ANALYSETTE 22 model with an inverse Fourier optic and the measuring cell position for: b) small particles, c) large particles [18]

The structural changes in copper and MA powders were studied by X-ray powder diffraction (XRPD) analysis which was performed by Rigaku Ultima IV equipment with CuKα Ni filtered radiation. XRPD patterns were scanned in 2θ range from 30° to 80°, at scan rate of 2°/min. Particle morphology changes of mechanical alloying powders were analysed using JEOL-JSM 5800LV scanning electron microscopy (SEM). The particle size distribution of mechanical alloying powders were determined by advanced laser nanoparticle sizer ANALYSETTE 22 NanoTec plus. The main principle of laser diffraction method is based on laser diffraction caused by particle presence. When particle is exposed to laser beam, the light diffraction occurs which resulting with characteristic, ring-shaped intensity distribution behind the sample which is measured by a specially shaped detector. The Fritsch GmbH was the first company which used an inverse Fourier optic for the particle size determination [17,18] and the first model was the ANALYSETTE 22 (Fig. 3a). The spacing of these occurred rings is used for calculation of particle size: the small particles cause widely spaced characteristic rings (Fig. 3b) while large particles produce more closely located rings (Fig. 3c). Fraunhofer diffraction method was used for calculation copper matrix composite particle diameters [18]. After detection and calculation of particle size (equivalent diameter), the results are presented as graphic diagrams.

The overall data is arranged according to the geometric dimension of the particle and plotted to the x-axis of coordinate system [18]. The components that are associated with the size of the individual elements and indicate the shares of individual particles within the overall distribution are depicted along the y-axis. The cumulative curve of the distribution particle size $Q_r$ (x) shows a standardised total quantity of all particles with equivalent diameters less than or equal to x. Parameter $q_r$ (x) shows the particle size range i.e. value is between $x_1$ and $x_2$.

## 3. Results and discussion

The results obtained by X-ray powder diffraction show that during mechanical alloying *in situ* forming of $ZrB_2$ particles in copper matrix is not possible (Fig. 4). Heowever, under the influence of high impact forces due collision ball-powder-ball mechanical activation of alloying elements and their penetration in the copper matrix occurs. In the first few hours of mechanical alloying the biggest changes occurs in the particle size of the starting powder mixture due to differences in the initial particle size and high impact forces in collision with balls. For powder mixture of Cu-Zr-B the biggest changes occur in first 10 hours of mechanical alloying (Fig. 5). Initially, the particles are plastic and can be easily deform. Brittle Zr particles are smaller and more fracture preferable compared to ductile copper particles, which can cause the insertion of Zr particles between balls and Cu particles resulting in copper particle fracture. The particle fracture leads to a reduction of the diameter and with the extension of the mechanical alloying time, balance of all three processes (deformation, fracture and cold welding) are occurred, resulting in more uniform distribution and particle form (Fig. 6) [19-21]. Ditto, all above mentioned fact provides forming of nanoparticles which were detected after 1 h of mechanical alloying (Fig. 6). After 20 hours of mechanical alloying, cold welding becomes dominant process which results in increasing the size of the powder particles and changing their shape.

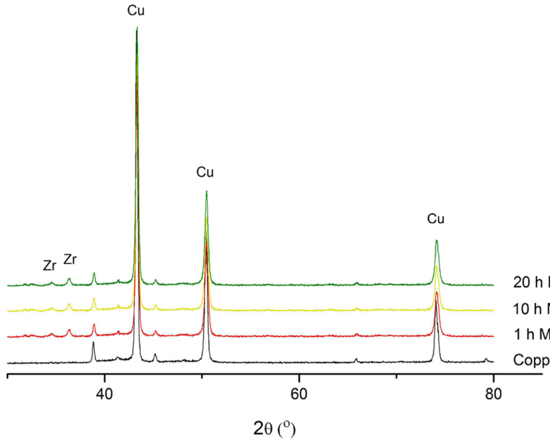


Fig. 4. XRPD patterns of copper and MA powders

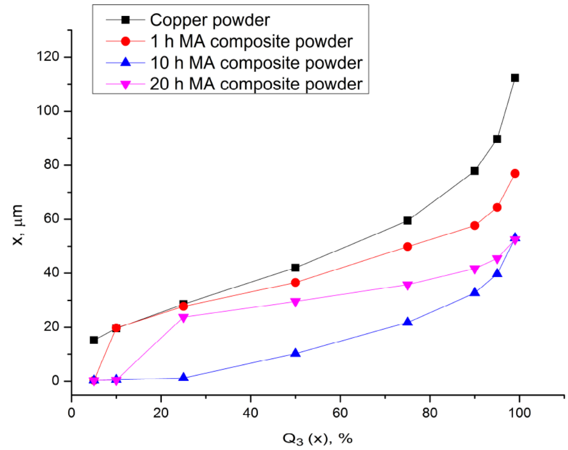


Fig. 5. Particle size distribution of copper matrix composites

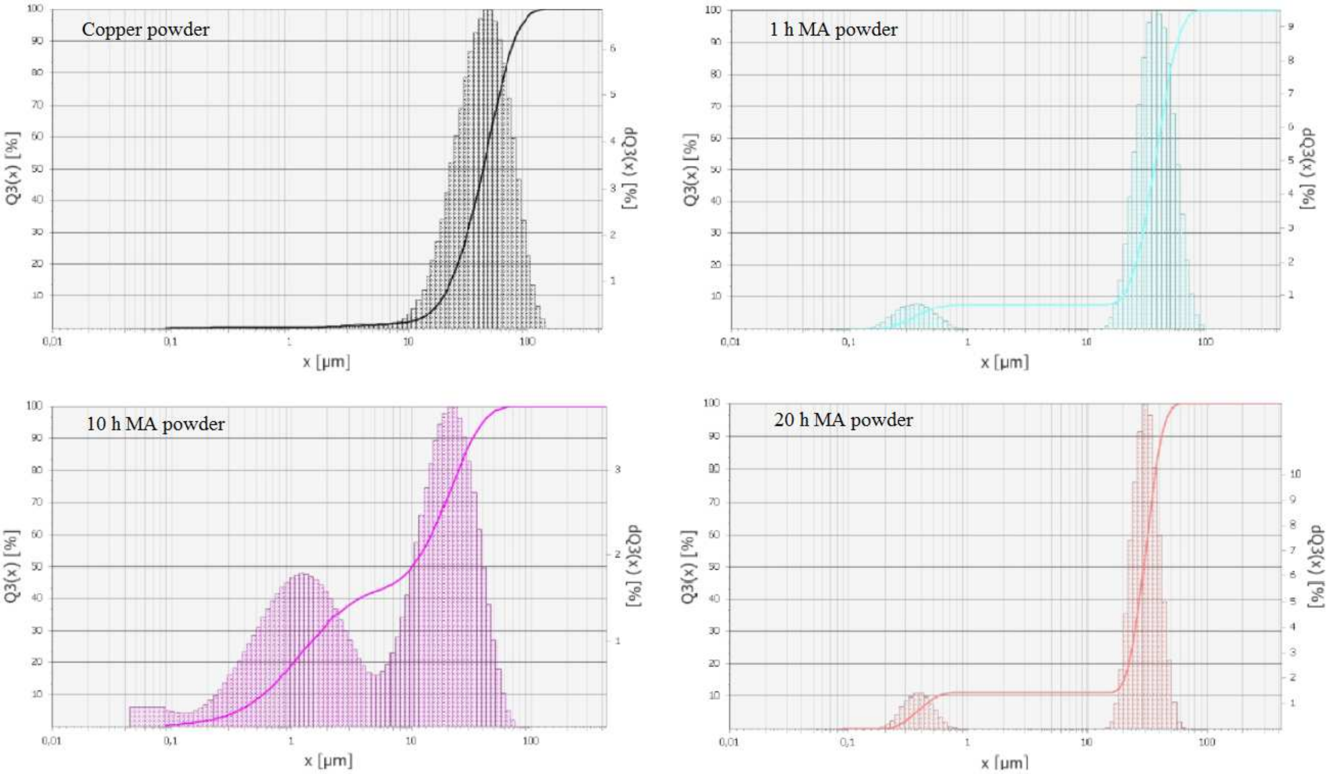


Fig. 6. Graphic diagrams of copper matrix composite particle size distribution obtained by laser nanoparticle sizer

Microphotographs of pure copper and mechanically alloyed powders for 1, 10 and 20 hours are presented in Fig. 7. Particles of pure copper (Fig. 7a) has dendritic structure and after 1 hour of mechanical (Fig. 7b) alloying dendrites become rounded and lamellar structure which is characteristic for MA powders can be noticed. After 1 hour of mechanical alloying, and with increasing mechanical alloying time, morphology of copper particles are rapidly changing from irregular shapes to more flat, plate-like form (Fig. 7c and 7d). The particles of starting powders have received flat shape due to deformation caused by high pressures at the interface between particles and balls.

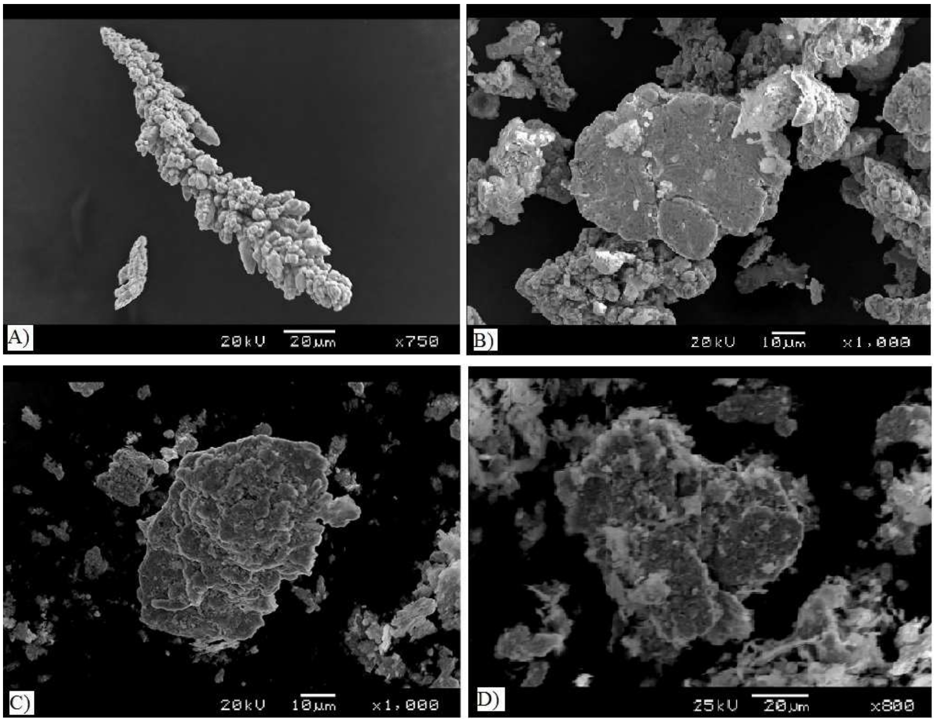


Fig. 7. SEM microphotographs: A) copper powder, B) 1 h MA powder, C) 10 h MA powder and D) 10 h MA powder of copper matrix composites

Comparing microphotographs for 1 h and 10 h of mechanical alloying it can be noted that after 1 h only some particles have lost their original shape, but after 10 h all particles have modified the initial morphology which is confirmed by the sharp decline in particle sizes (particle size distribution curve, Fig. 4). Curve which shows particle size distribution after 20 h of mechanical alloying indicates that process of cold welding is dominant and particle diameter has increased.

Previous studies [22,23] showed that mechanical alloying time has a great influence on properties of copper matrix composites obtained by powder metallurgy technique. Therefore, proper selection and optimization of mechanical alloying parameters may provide control and reliable prediction of desirable properties of final copper matrix composites. However, results presented in this study are very important for further studies of these novel composite materials. Identification of nanoparticles and their presence during mechanical alloying of copper matrix composite materials point out the influence of added hard particles of zirconium and boron on copper matrix and it provides better understanding of properties of these composite materials. Also, obtained results can be used as inputs in particle-based computational models which can provide prediction of desirable mechanical and/or physical properties of composite materials.

## 4. Conclusions

Results obtained by analysing influence of mechanical alloying time on particle size and morphology show the presence of nanoparticles which is novelty for copper matrix composite with addition of zirconium and boron. Advanced laser nanoparticle sizer has proved to be good selection for analysing these kind of composite materials. Analysis of particle shape and size identify dominant processes which occur at specific time during mechanical alloying of copper matrix with addition of zirconium and boron. Also, it was determine that after 10 hours of mechanical alloying balance of all three processes: deformation, fracture and cold welding, is achieved. After 20 hours cold welding process is dominant which results in characteristic lamellar structure of powders. Obtained results will be very helpful for further optimization of mechanical alloying of copper matrix and encourages future similar research in order to expand knowledge about the influence of mechanical alloying parameters on final material properties.

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## Additional information

Selected issues related to this paper are planned to be presented at the 22nd Winter International Scientific Conference on Achievements in Mechanical and Materials Engineering Winter-AMME'2015 in the framework of the Bidisciplinary Occasional Scientific Session BOSS'2015 celebrating the 10th anniversary of the foundation of the Association of Computational Materials Science and Surface Engineering and the World Academy of Materials and Manufacturing Engineering and of the foundation of the Worldwide Journal of Achievements in Materials and Manufacturing Engineering.

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