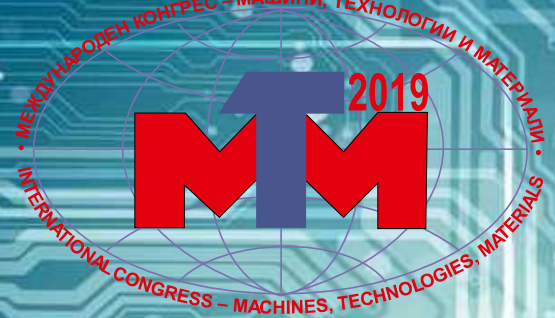
XVI INTERNATIONAL SCIENTIFIC CONGRESS

SUMMER SESSION

11 - 14.09.2019, VARNA, BULGARIA




# MACHINES TECHNOLOGIES MATERIALS 2019

# PROCEEDINGS

## VOLUME III
## MATERIALS MACHINES & INDUSTRIAL DESIGN

ISSN 2535-0021 (Print)
ISSN 2535-003X (Online)

ORGANIZER
SCIENTIFIC-TECHNICAL UNION OF MECHANICAL ENGINEERING
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**YEAR III, ISSUE 3 (14), SOFIA, BULGARIA 2019**

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PUBLISHER:

**SCIENTIFIC TECHNICAL UNION OF MECHANICAL ENGINEERING**

108, Rakovski Str., 1000 Sofia, Bulgaria
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# C O N T E N T S

## *MATERIALS.*

**METHOD OF PERMEABLE ELEMENTS FOR SIMULATION OF POWDER METALS FORMING PROCESSES**
Dr.Sc. Bagliuk G.A. ..... 226

**PROCESSING OF BIMETALS IN THE ELECTROLYTE PLASMA**
Prof. Dr. Eng. Kaputkin D., Prof. Dr. Eng. Duradji V., Ass. Prof. Dr. Eng. Kaputkina N. ..... 230

**THE EFFECT OF BALL MILLING SPEED ON THE PENETRATION OF Cr AND Zr INTO Cu FOR PRODUCTION OF Cu1CrZr ALLOY POWDER**
Prof. Dr. Kul M., Assoc. Prof. Dr. Özer A., Dr. Öksüz K.E., Kurt N. ..... 233

**Al MATRIX FGM HAVING 5, 15 AND 25 % B4Cp REINFORCED LAYERS**
Prof. Dr. H. Erdem Camurlu ..... 238

**FINITE ELEMENT SIMULATION ON TENSILE CREEP BEHAVIOUR OF UNDERGROUND SUPPORT LINER**
Guner D., Ozturk H. ..... 241

**FAILURE CHARACTERIZATION OF ROCK ANCHOR BOLTS BY THERMAL CHANGE DETECTION UNDER TENSILE STRENGTH TESTING**
Asst. Prof. Dr. Mustafa Erkayaoğlu, Alper Kırmacı ..... 245

**FEATURES OF WEAR OF RAILWAY WHEELS TREAD**
Prof.. Dr. Sci. Gubenko S. ..... 249

**THE PHENOMENON OF REVERSION IN AGING MAGNESIUM ALLOYS WITH THE RARE-EARTH METALS**
Prof. Rokhlin L.L., PhD. Dobatkina T.V., Tarytina I.E., PhD. Lukyanova E.A. ..... 253

**EFFECT OF AGING ON ZAMAK 3**
H. Erdem Camurlu, İsmail Aktuna ..... 256

**FORMATION OF ZrB2-AlN POWDER MIXTURE BY SELF-PROPAGATING HIGH-TEMPERATURE SYNTHESIS**
H. Erdem Camurlu ..... 259

**MATERIAL-SCIENCE ASPECTS OF FORMATION AND EVOLUTION OF FLAWS WHICH DEFINE THE RESOURCE EXPLOITATION OF ALUMINUM STRUCTURES OF AIRPLANES**
Prof. Dr. Chernega S., phd. Abolihina H.,phd. Grinenko K. ..... 262

**PREPARATION AND CHARACTERIAZATION OF MANGANESE OXIDES AND BIRNESITES, APLIED AS ELEKTRODE MATIRIALS IN ION BATTERIES**
гл. ас. д-р Златанова З, Кирева М. ..... 264

**EFFECT OF STRUCTURE AND TEXTURE ON THE MECHANICAL CHARACTERISTICS OF MAGNESIUM ALLOYS PROCESSED BY EQUAL-CHANNEL ANGULAR PRESSING**
PhD Martynenko N.S., Tokar A.A., PhD. Serebryany V.N., PhD. Prosvirnin D.V., Prof., Dr. Sci. Terentiev V.F., Prof., Dr. Sci. Raab G.I., Prof., Dr. Sci. Dobatkin S.V., Prof., PhD Estrin Yu.Z. ..... 267

**THE STRENGTHENING AND RENWALING OF DAMAGER REINFORCED CJNCRETE STRUCTURES BY HRESTRESSED FIBER REINFORCTD PLASTICS**
Prof. Dr. Aliy Bespayev , Eng. Ulan Kuralov, Eng. Ulan Altigenov ..... 271

**ENERGY HEAT EXCHANGE IN THE ZONE OF CONTACT OF THE PROBE OF AN ATOMIC FORCE MICROSCOPE WITH THE SURFACE UNDER STUDY**
Andriienko O., Ralchenko S., Doctor of Science Bondarenko M., PhD Bondarenko Yu. ..... 275

**PROPERTIES OF NANODIAMONDS OF INDUSTRIAL DETONATION SYNTHESIS**
Doctor of Technical Sciences, Professor, Dean of the Special Engineering Faculty E. A. Petrov; Postgraduate Student, SC Federal Research and Production Center “Altai” A. A. Kolesova; Director, Research and Production Enterprise "SIDAL" A. B. Pribavkin ..... 280

**PECULIARITIES OF THE INTERACTION OF A HIGHLY CARBONIZED FERROCHROME WITH IRON DURING SINTERING OF 65% Fe-35% FH800 COMPOSITE**
Dr.Sc. Maslyuk V., Eng. Karaimchuk E., PhD. Gripachevsky O., Dr.Sc. Bagliuk G., R.As. Sytnyk I. ..... 283

**COMPUTER MODELING OF INFLUENCE OF PREVIOUS DEFORMATION DEGREE AND STRAIN RATE ON CARBONITRIDES PRECIPITATION KINETICS IN LOW-CARBON MICRO-ALLOYED STEEL**
Ph.D. Kaverynskyi V., Eng. Sukhenko Z., Prof., Dr. Sc. (Eng.) Bagliuk G. ..... 286

# THE EFFECT OF BALL MILLING SPEED ON THE PENETRATION OF Cr AND Zr INTO Cu FOR PRODUCTION OF Cu1CrZr ALLOY POWDER

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***Abstract:*** *The copper alloys are preferred for their superior mechanical properties combined with high electrical conductivity for contact material applications. These properties should be a very good combination of high mechanical strength and enough electrical conduction properties as well as high thermal conductivity. To establish the electrical and thermal conductivity, the low resistive materials such as copper and their alloys to be chosen is a must. Copper can be alloyed with Cr and Zr in very low amounts down to 1 wt% and 0.15 wt%, respectively. These amounts can be found from phase diagrams of interest. The planetary ball milling was chosen as being a unique technique for powder processing to obtain mechanochemical activation and mixing of powders prior to sintering. The ball milling jar was $ZrO_2$ and ball to powder ratio was varied as 1:1, 5:1, and 10:1. The ball milling rotation was ranged from 400 and 500 rpm for different durations to be investigated. The SEM photos showed the Cr and Zr penetration onto Cu powders transformed to flakes of different sizes. The elemental composition maps showed the better and finer distribution of alloying elements at 500 rpm than 400 rpm.*

***KEYWORDS***: CuCr1Zr ALLOY, BALL MILLING, PENETRATION INTO Cu.

## 1. Introduction

Hard copper alloys can be industrially classified into three main groups; these are 1) aluminum bronzes having high strength and wear resistance, 2) beryllium containing, 3) beryllium free alloys for the applications that need electrical conductivity. But, during the last decade, the negative effect of beryllium on environment and health has become an issue and the alloy formation with beryllium was left. Instead of these alloys, a known CuCr1Zr alloys has begun to be used [1].

From an industrial view point, copper is the first mentioned and very well known as high electrical conductive material with high abundance and being cheap. But the pure copper has very low hardness (49 HV) and yield strength (54 MPa) which makes the use of it impossible in point arc welding process. This problem is overcome by alloying the Cu. But many strengthening processes to improve the mechanical strength as solid solution strengthening, grain refinement, alloying and dispersion hardening are inversely proportional with electrical conductivity property. For instance, while the electrical conductivity decreases down to 5%-75% of IACS by alloying and plastic forming. The electrical conductivity of CuCr1Zr varies between 76-90%IACS but the hardness increases up to 156 HB. By this study a unique, comparative and systematical approach will be carried out to partially fulfill the literature [2, 3].

Composites are of the materials with a 3D network as matrix and a reinforcement material. The superior properties of both materials as a new material can be achieved together as hardness from ceramics and toughness from metals, etc. As very well known, the electrical conductivity of metals is inversely proportional with the increasing mechanical strength. Since the alloying elements produce local strain and stress fields around the lattice, the mean free path of electrons become smaller by scattering from atoms of impurity elements. The decreasing mean free path of electrons decreases the conductivity while increasing the resistance. The dislocation interruptions are also obstacles for electrons to decrease the conductivity while increasing the strength by interfering with dislocation movement. Besides, following the plastic forming or strength improvement, the aging may recover the conductivity while decreasing the strength, but to a small extent. Cu, Cr and Zr will be planetary ball milled in appropriate weight ratios to produce CuCr1Zr alloy. Also, after every process, the mechanical-electrical and thermal properties will be measured continuously to monitor the changes. The effect of process parameters on the physical properties will be investigated. The hard copper alloys are used in the application areas that need high electrical and thermal conduction and mechanical strength as point electrode arc welding. The increasing energy demand worldwide and decreasing source of energy, by increasing energy prices makes the industry using intense energy input to develop high electrical-thermal stable and conductive as well as mechanically strong materials as automotive, panels, radiator fabrication systems.

As the pre-evolutionary result, the first part of the project, which is production of CuCr1Zr powder alloy by mechanic activation, was presented.

## 2. Materials and methods

Pure spherical Cu (purity 99.8%, Dv50 25 μm), Cr (purity 99.9%, Dv50 3 μm) and $ZrH_2$ (purity 99.8%, Dv50 5 μm) powders were provided by Alfa Aesar as seen from Fig 1(a-c). The size of the metallic and compound powders was determined by FEG-SEM (Tescan Mira3 XMU, Czechia). XRD analyzes were performed using the RIGAKU D-MAX 2200 X-ray diffractometer. Microstructural analysis (grain structure and grain shape), phase formation and surface morphology of the produced CuCr1Zr alloy powders were carried out by using FEG-SEM. Sonics VCX750 ultrasonic horn was used to produce homogeneous CuCr1Zr powder mixtures. The powders were milled with Fritsch pulverisette 7 Premium line planetary milling device.

Since the $ZrH_2$ particle size is too coarse to be used in the studies, it has been subjected to a separate size reduction process. $ZrH_2$ powder with coarse particle size was subjected to wet milling in ethanol for 24 h and the particle size was reduced down to 3±1 microns (Fig. 1d).

Cu, Zr and Cr powders were dispersed in the separate containers using ethanol by ultrasonic homogenizer for 10 minutes. The separately prepared powders were then mixed in a clean beaker and subjected to ultrasonic stirring for 10 minutes using an 80% power at 5 s pulse durations. The prepared powders were placed in 80 ml zirconium oxide grinding jars. After adding zirconium oxide beads to the grinder jar in the determined proportions, the amount of ethyl alcohol was adjusted to 30 ml and the safety locks of the grinder jars were closed into the planetary mill. The milled powder in the planetary mill was separated from the balls using a clean wet sieve of -38 microns and then the powder-ethanol mixture continuously stirred rigorously at maximum of 60 °C to evaporate the ethanol. After that milling, SEM photographs were analyzed.

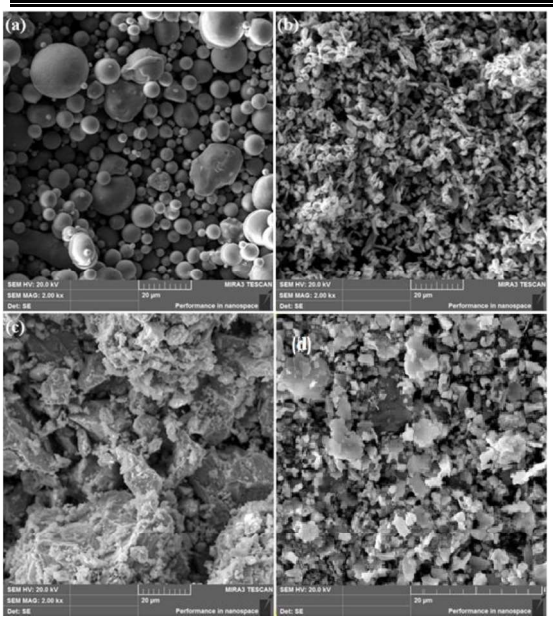



***Fig. 1.*** *The SEM images of (a) Cu, (b) Cr, (c) $ZrH_2$, (d) after ball milling of (c).*

## 3. Results and discussions

### 3.1. Preliminary Experiments of Mechanical Alloying

The centrifugal forces created by the movement of the rotating carrier disk and the movements of the chambers rotating about its axis in the device impact the balls and the metal particles. As seen in Fig. 2, the centrifugal forces act in similar and opposite directions since the chambers and the disk on the disk rotate in opposite directions. This causes friction by moving the balls downward on the inner surface of the chamber in which they are located. The friction effect continues when the metal particles and balls circulate freely in the cup jar and strike the inner surfaces of the jar.

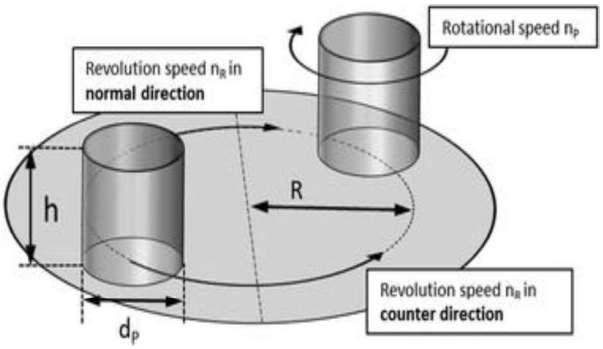



***Fig. 2.*** *Demonstration of movement direction of discs and chambers in planetary mill.*

Cu1Cr0.15Zr composition (1% Cr, 0.15% Zr and 98.85% Cu) was kept constant in order to determine mechanical alloying conditions in mechanical alloying.

It is contemplated that the embedding of Cr and Zr particles into Cu grains can be quantitatively analyzed by powder shape change from spherical to flakes and stratification. In these experiments, it was determined that Cu grains were expanded as elliptical or flake by deformation. Increased Cu flakes indicate increased collision statistics and increased stress intensity. Fig. 3(a) shows the Cr and Zr distribution in Cu powders before milling while Fig. 3(b) indicates the distribution of finer Cr and Zr powders into Cu. Cr and Zr were not homogeneously distributed and embedded into Cu particles as shown the SEM images (Fig. 3-a). But they embed into Cu particles as finer particles after milling as shown the SEM images in the Fig. 3-b.

The main factor that indicates Cr and Zr are embedded into copper particles is that the spherical copper particles deform in the plate until the smallest distance between them remains. Thus, Cr and Zr impregnation on growing Cu flakes can be observed more effectively. For this reason, it has been tried to determine to what extent the mechanical alloying is successful by emphasizing the rate at which Cu powders were flaked, stratified and severely deformed. In order to convert the success of mechanical alloying into analytical data, it was decided to determine by conversion from sphericity to ellipse (CFSTE) (or deviation from sphericity) using linear intercept method. The details of this method are given below in Fig. 4 with an example.

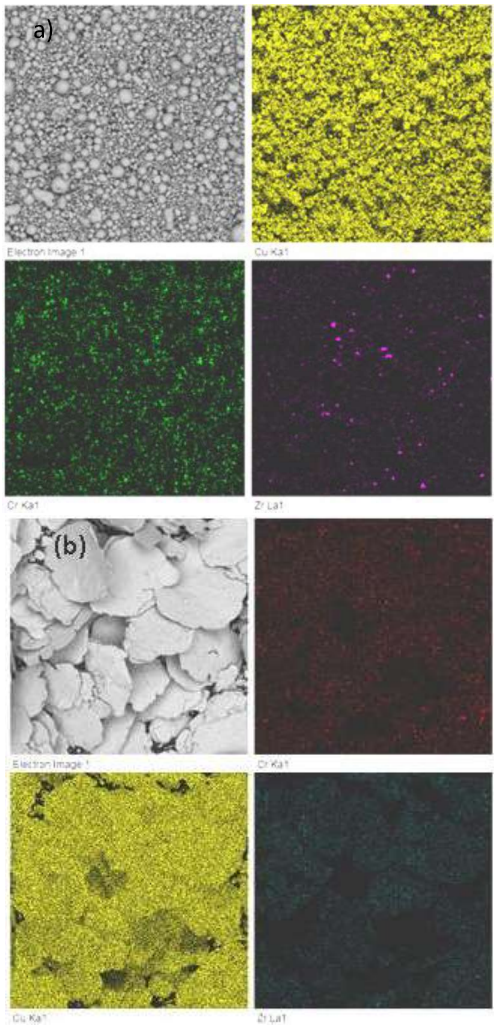



***Fig. 3.*** *SEM-EDX Elemental distribution maps a) before milling, b) after milling.*

### 3.2. Percent Conversion from Sphericity to Ellipse Calculation

CFSTE ratio = ((X-axis length of ellipse/Y-axis length of ellipse)/1.5)*100

…… (1)

For the powder CFSTE measurement of powder Cu1Cr0.15Zr alloys; the X-axis length and Y-axis length of each ellipse particle were measured in microns (Table 1) using Nitro Pro from the SEM photographs (Fig. 4). The length obtained in micrometers was then divided by the bigger value over the smaller value to determine the CFSTE rate of each powder. For example, the lengths of the 32 powders on the X and Y axis of the SEM photograph given in Fig. 4 were measured with the help of the drawn squares and entered into

***Table 1.*** *The data used to determine the CFSTE rate of 32 grains in Figure 4.*

| Grain No | X (µm) | Y (µm) | X/Y or Y/X | Grain No | X (µm) | Y (µm) | X/Y or Y/X | Grain No | X (µm) | Y (µm) | X/Y or Y/X |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **1** | 19.5 | 17.5 | 1.114286 | **12** | 25 | 20 | 1.25 | **23** | 25 | 22.5 | 1.111111 |
| **2** | 65 | 58 | 1.12069 | **13** | 35 | 47.5 | 1.357143 | **24** | 19.5 | 17.5 | 1.114286 |
| **3** | 15 | 15 | 1 | **14** | 70 | 40 | 1.75 | **25** | 65 | 58 | 1.12069 |
| **4** | 32.5 | 30 | 1.083333 | **15** | 62.5 | 45 | 1.388889 | **26** | 15 | 15 | 1 |
| **5** | 74 | 62.5 | 1.184 | **16** | 69 | 75 | 1.086957 | **27** | 20 | 22.5 | 1.125 |
| **6** | 90 | 75 | 1.2 | **17** | 35 | 25 | 1.4 | **28** | 15 | 20 | 1.333333 |
| **7** | 75 | 60 | 1.25 | **18** | 82.5 | 75 | 1.1 | **29** | 27.5 | 35 | 1.272727 |
| **8** | 30 | 27.5 | 1.090909 | **19** | 51.5 | 35 | 1.471429 | **30** | 15 | 15 | 1 |
| **9** | 35 | 30 | 1.166667 | **20** | 47.5 | 35 | 1.357143 | **31** | 21.5 | 32.5 | 1.511628 |
| **10** | 75 | 87.5 | 1.166667 | **21** | 75 | 67 | 1.119403 | **32** | 27.5 | 29.5 | 1.072727 |
| **11** | 80 | 65 | 1.230769 | **22** | 47.5 | 40 | 1.1875 | | | | |
| **X/Y Average = 1,219266 ; Standard deviation ± 0,161891** | | | | | | | | | | | |

MS Excel as shown in Table 1. From these values, CFSTE rates (X/Y or Y/X values) of these 32 powders were calculated. Then, the average CFSTE ratio was determined for this SEM photograph of the sample by taking the arithmetic mean of the CFSTE rates calculated for these 32 values. Then, the same method was repeated for an SEM photograph of the same sample on a larger scale and the arithmetic average of the two ellipse rates obtained from these two photos by calculating the ellipse value of the sample using Equation 1. As mentioned previously, this value is considered as the successful measure of mechanical alloying. As the method was summarized from SEM photograph of a sample in Fig. 4, the lengths of 32 X and Y axes were measured (Table 1) and the average ellipse rate for this photograph was determined as 1.219266 from the above-described method. Then, the same process was repeated for 50 units in another photograph of the same sample at larger magnification and the ellipse rate was calculated as 1.22894 and the CFSTE rate of the sample was found as 1.224103 by taking the arithmetic average of these values. Using this value, the ratio of ellipse in mechanical alloying was determined as = (1.224103 / 1.5) * 100 = 81.61. At the end of each mechanical alloy test, at least 2 SEM photographs were obtained.

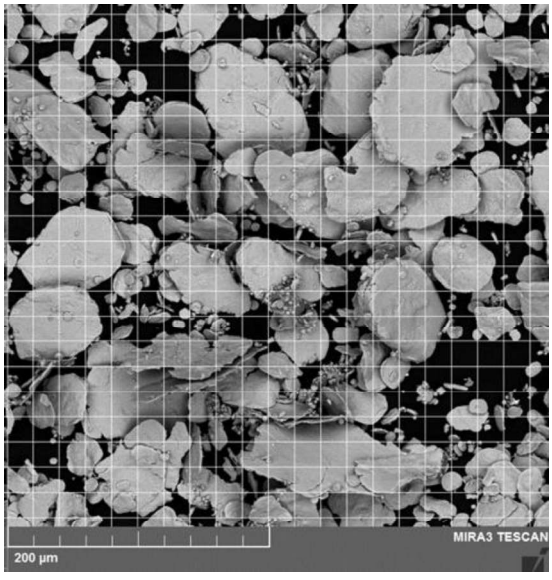



***Fig. 4.*** *SEM photo of a mechanically alloyed sample used in the calculation of CFSTE %.*

Chemical analysis of CuCrZr powder alloy by EDX after mechanical alloying showed that Zr contents of these powders increased. Since the planetary mill used in mechanical alloying was used by the zirconia mortar and the zirconia balls, it was decided to follow the Zr content of powder alloy in mechanical alloying studies and after each experiment. SEM-EDX chemical analysis of the ground powders was carried out to determine the Zr content of the powder mixtures and to investigate the effect of alloying parameters on the Zr content of the powder alloy from zirconia mortar and ball. This increase in Zr content has been seen as a problem, since Zr passing from mortar or ball to powder alloy is in the oxide phase, which will reduce the electrical conductivity of the final product. Therefore, in the selection of alloying parameters, has been decided to choose the ones with the Zr content of the alloyed powder 0.15 wt% or the closest. Hence the initial Zr content of the powder mixture in these studies was 0.15% and this value was held constant in all experiments in this study. As a result, the effect of the parameters on the CFSTE ratio and Zr content of alloy powders was investigated and optimum mechanical alloying parameters were determined.

### *3.3. Milling at 400 rpm*

Fig. 5 shows SEM (BSE) images of the powder mixture milled in a planetary mill for 1 hour at 400 rpm. Accordingly, the amount of deformation increases at 400 rpm with increasing ball to powder ratio. The increase in the amount of deformation was characterized by flattening of spherical Cu powders. With increasing ball to powder ratio, collision statistics and stress intensity increase, so that deformation imposed on Cu increases. However, as shown in SEM photo Fig 5(a), spherical powders are not suitable for Cr and Zr penetration, as they have a large surface area but do not tend to be flake superficially. It is thought that by using the deformation ability of Cu, increasing plating, ex-foliation and hard and brittle powders such as Cr and Zr will be broken and fragmented and buried on Cu surface. As a result of planetary milling for 1 hour at 400 rpm is not sufficient for embedding of Cr and Zr into Cu powders for all ball/powder ratios.

***Table 2.*** *Effect of ball/powder ratio on CFSTE rate and Zr content of powder mixture. (1 h of milling at 400 rpm).*

| Ball/Powder ratio | **%** CFSTE ratio | Zr % |
|---|---|---|
| 1/1 | 80,10 | 0,261 |
| 5/1 | 82,03 | 0,187 |
| 10/1 | 78,61 | 0,158 |
| 20/1 | 75,88 | 0,129 |

### *3.4. Milling at 500 rpm*

In Figure 6, SEM images of alloy powders milled at 500 rpm for 1 hour are available. As can be seen, even in the 1: 1 ball/powder ratio, effective results were found better than 400 rpm, and the deformation intensity of the powders increased and the foliation was observed. However, again at this rate and within 1 hour, it was found that there were still powders close to the sphere in the mixture, so that there was not enough plate surface for Cr and Zr penetration.

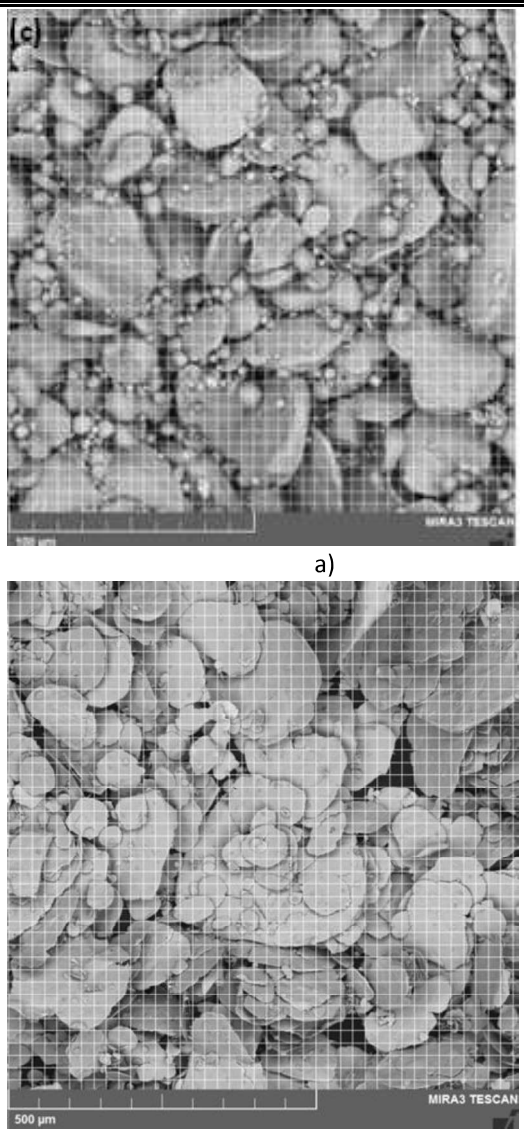



***Fig. 5.*** *SEM images of a) 10/1, b) 20/1 ball/powder ratios.*

Table 3 shows the effects of ball/powder ratio on CFSTE ratio and Zr wt% determined by EDX analysis. The increase in ellipse shows that the deformation increases, the platened powder size becomes more brittle and tends to break, as well as thickening of the plates by stratification. One can conclude that a value of 1.3182 (87.88%) is one of the upper boundaries of ellipse as to be said that the ellipse value is close to 1.5 and the exact ellipse is the limit of deformation. $ZrO_2$ transport from balls to the powder mixture which seems to increase with the increasing ball/powder ratio is also thought to be increased with the statistics, number and intensity of collisions. According to these results, milling at 5/1 ball/powder ratio 500 rpm for 1 hour or milling at 1/1 ball/powder ratio 500 rpm for 5 hours is suitable for Cr and Zr penetration.

***Table 3.*** *Effect of ball/powder ratio on CFSTE rate and Zr content of powder mixture.*

| 1 hour milling at 500 rpm | | |
|---|---|---|
| Ball/Powder ratio | **%** CFSTE ratio | Zr % |
| 1/1 | 86.73 | 0.104 |
| 5/1 | 83.17 | 0.147 |
| 10/1 | 82.21 | 0.185 |
| 5 hour milling at 500 rpm | | |
| 1/1 | 82.05 | 0.167 |
| 5/1 | 86.19 | 0.257 |
| 10/1 | 87.88 | 0.309 |

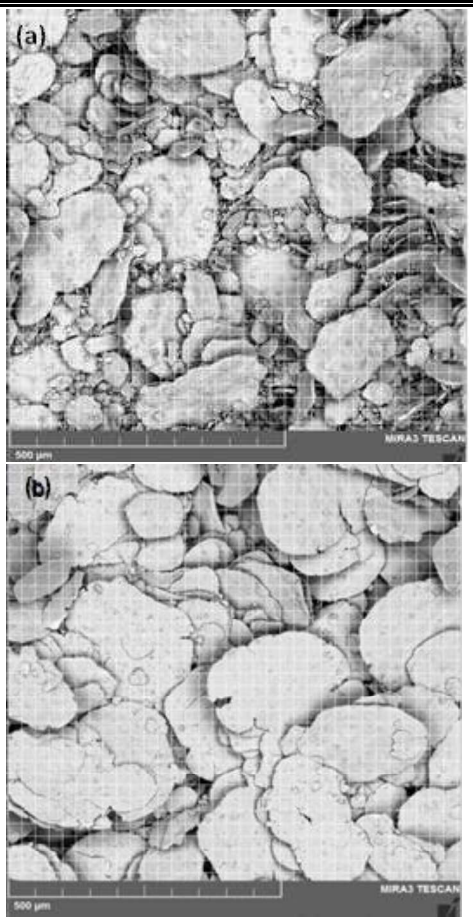



***Fig. 6.*** *SEM images of a) 5/1, b) 10/1 ball/powder ratio.*

Fig. 7 shows the plating, Cr-Zr penetration and folding of Cu powder at a speed of 500 rpm and different durations. At low deformation times, the layers have just begun to overlap, but surface cracks have also occurred, and the penetrating Cr is partially retained in the grain shape, and the Cu surface is still smooth. At increasing speed, it is seen that the smoothness of the Cu surface is deteriorated and the shape of the Cu powder becomes close to the circle due to deformation. The effect of increased deformation on the stratification of the Cu plate can also be observed as folding or tearing. As can be understood from this, as the deformation increases, Cu can be folded and deposited on itself, leading to agglomeration of Cr and Zr penetration between them and subsequent plate breakage.

## 4. Conclusions

The $ZrO_2$ content of the powder mixture increases with increasing ball / powder ratio. Therefore, high ball / dust ratio should be avoided, especially at high speeds and long grinding times. Otherwise, the electrical conductivity of the composite material will decrease due to $ZrO_2$. Milling at 5/1 ball/powder ratio, 500 rpm for 1 hour or milling at 1/1 ball/powder ratio, 500 rpm for 5 hours can be suitable for Cr and Zr penetration into Cu particles.

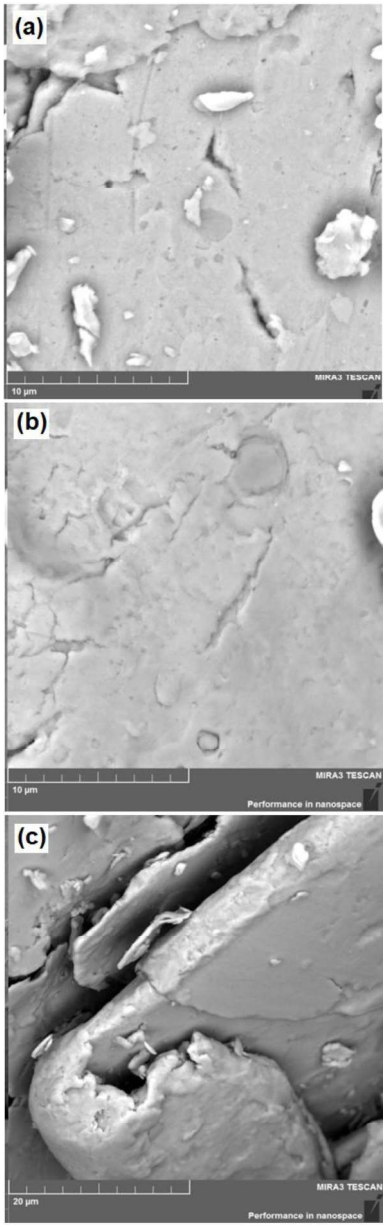



***Fig.7.*** *(a) Cr-Zr embedding morphology in powder rotated at 10: 1 ball / powder ratio at 500 rpm for 3 hours, (b) Cr-Zr embedding morphology in powder rotated at 10: 1 ball / powder ratio at 500 rpm, (c) stratification and excessive deformation of the plates of powder rotated with a 10: 1 ball / powder ratio at 500 rpm for 5 hours.*

**Acknowledgments**

This research is funded by **TUBITAK** (The Scientific and Technological Research Council of Turkey) under the grant number #117M560.

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