The Effect of Rolling Temperature and Rolling Ratio on Hardness and TRS Boron Carbide 15% (B4C) Reinforcement Copper Matrix Composite

Özkan ESKI¹, Hasan ALSead Ibrahim ENBIA²

¹,²Kastamonu University, Engineering Faculty, Kastamonu University, Institute of Science, Kuzeykent Campus, Kastamonu - Turkey

Email: ozkanesk[at]kastamonu.edu.tr,
Email: Enbayahasen[at]gmail.com

Abstract: Copper is one of the most commonly used metal for industrial purposes due to high thermal and electrical conductivity, ductility etc. but it has poor abrasion resistance. For improving these poor properties scientific researches goes on. Reinforcing by adding ceramics as a reinforcement material like SiC, B₄C is a commonly used method for better mechanical properties. Copper powders mixed with ceramic particles hot pressed or cold pressed then sintered to achieve high hardness and good resistance for abrasion wear. In this study 15% wt, boroncarbide, 90 µm grain size, added copper powder mixed mechanically then cold pressed under 15 MPa and sintered at 850 °C in argon atmosphere for 120 minutes. Metal matrix composites hot rolled at three different rolling temperature (600 °C, 700 °C and 800°C) and four rolling ratio (10%, 20%, 30% and 40%). Mechanical properties like hardness and Transverse Rupture Strength (TRS) of composites measured, compared depending on rolling temperature and rolling ratio. SEM and EDS applied for microstructural analysis; it is found that distribution of B₄C is uniform. Rolling at 600 °C increased both hardness and TRS for all rolling ratio.

Keywords: Powder metallurgy, B₄C, Hot Rolling, Cold Pressing

1. Introduction

A composite material is produced by combining two or more materials to create a unique combination of properties, frequently ones with radically distinct characteristics [1,2]. The two components combine to give the composite its special qualities. Typically, the continuous or matrix component contains the reinforcing component [3]. The composite is known as a metal - matrix composite (MCMs) when the matrix is made of metal. Particles, whiskers, short fibers or continuous fibers are frequently used as reinforcement in MMCs. Nowadays composite material’s main advantage against conventional materials are both light weight and high strength. A new material that precisely satisfies the requirements of a given application can be created by selecting the right matrix and reinforcement material combination [4]. Due to its excellent mechanical qualities and light weight, metal matrix composites have been used in a variety of industries, including automotive, biotechnology, aerospace, defense, and energy [5]. The main elements that allow the properties of metal matrix composites to improve are those of the matrix, the reinforcement, and the reinforcement - matrix interface. SiC, Al₂O₃, TiC, and B₄C are excellent reinforcement materials used in the creation of matrix composites that resemble copper [6]. In the literature, the powder metallurgy process is frequently used and plays a significant role in the fusion of alloys and metal matrix composites [7].

Copper - based ceramic particulate - reinforced metal matrix composites (CMCs) have been gaining much attention owing to their good mechanical, thermal, and tribological properties. When the matrix needs to maintain good wear resistance without losing its ability to conduct heat and electricity, CMMCs are used [8]. The ductility and toughness of CMMC are reduced when hard, non-deformable ceramic particles are added to matrix alloys. The lifespan of components in various applications is influenced by their surface characteristics. As a result, it is reasonable to change the component's surface by adding ceramic reinforcement while preserving the ductility and toughness of the inner matrix. Surface metal matrix composite (SMMC) is the name of the modified surface dispersion - strengthened composite layer [9].

Copper (Cu) is an extensively used metal due to its exceptional qualities, including resistance to corrosion, ductility, formability, high thermal and electrical conductivity, deformability [1]. Cu is especially used in applications where high electrical and thermal conductivity is required because of its outstanding properties [8]. Cu and its alloys' principal flaws are their inadequate wear resistance and low strength [3]. Cu has been studied for a number of uses, but approximately 60% of them involve electrical conductivity [1]. Cu is relatively soft when it is commercially pure, unlike other highly conductive metals. To strengthen metallic materials, five main techniques are used: tension, dispersion hardening, sedimentation, solid solution, and grain boundary [8]. Sedimentation and solid solution are related to the addition of the metal to particular alloying elements, which can improve the mechanical properties of the metal but can significantly reduce its electrical conductivity [7]. On the other hand, the electrical conductivity of copper is marginally decreased by tension, dispersion hardening, and grain boundary methods.

Boron carbide is characterized by a unique combination of properties that make it a material of choice for a wide range
of engineering applications. Due to its extreme abrasion resistance, boron carbide is used in abrasive powders and coatings, refractory applications due to its high melting point and thermal stability, ballistic applications due to its high hardness and low density, and as a neutron radiation absorbent in nuclear applications. Boron carbide is also a high - temperature semiconductor that may be applied in cutting - edge electronic applications [10]. The aim of this research is to investigate the effects of hot rolling temperature (600°C, 700°C, 800°C) and rolling ratio (10%, 20%, 30%, 40%) on Cu - B4C 15%wt. cold pressed and sintered at 850 °C sintered composite’s mechanical properties like hardness and Transverse Rupture Strength (TRS).

2. Experimental Procedures

Copper (Cu) powder, 40μm grain size and 99.9% pure and boron carbide (B4C) 99.5% purity and 90μm grain size used for producing samples. Samples cold pressed in dimensions 24mm x 10 mm x 5 mm under 15 MPa pressure in steel die, shown in fig.1.

![Figure 1: Sample](image1)

Copper (Cu) 85% wt. base material of the compound and boron carbide (B4C) 15%wt. reinforcing material placed in three - axis mixing machine and mixed for 60 minute at 45 rpm for homogenous distribution in structure. Produced green samples sintered at 850 °C in a sintering device for 90 minutes under argon atmosphere, as shown in fig 3.

![Figure 2: Sintering furnace](image2)

![Figure 3: Cycle of sintering temperature](image3)

The rolling mill, shown in Figure 4, used for hot rolling at 600°C, 700°C, 800°C and rolling ratios 10%, 20%, 30%, 40%. Hot rolling process carried out in the laboratories of Karabuk University. Roller’s diameter is 70 mm and roller speed 30 r/min, approximately 6.5 m/sec.

![Figure 4: Rolling machine](image4)

Table 1: Showing the final thickness of the samples after deferent rolling ratios

<table>
<thead>
<tr>
<th>Rolling reduction ratio</th>
<th>Final thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>5 mm</td>
</tr>
<tr>
<td>10%</td>
<td>4.5 mm</td>
</tr>
<tr>
<td>20%</td>
<td>4 mm</td>
</tr>
<tr>
<td>30%</td>
<td>3.5 mm</td>
</tr>
<tr>
<td>40%</td>
<td>3 mm</td>
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</tbody>
</table>

Produced and hot rolled samples grinded by using sand paper in different mesh size and polished by using diamond solution then rinsed with alcohol to prevent oxidation.

Density of Cu - B4C samples measured by using Archimedes principle. Hardness of the samples measured in 5 different points on surface under 62.5 kgf with a steel ball of 2.5 mm diameter by using BULUT BMS 300 OPBC full automatic Brinnel test machine then average hardness values considered as a hardness of the sample. To assess the
bonding quality and determine the transverse rupture strength (TRS) of the Cu - B4C, three - point bending tests were performed according to ASTM B528 - 83a by using a SHIMADZU universal tensile machine with a maximum capacity of 50 kN in Kastamonu University Laboratory, at 0.5 mm/min test speed. Scanning Electron Microscopy (SEM) imaging and EDS analysis performed in Kastamonu University Central Research Laboratory by using FEI QUANTA FEG 250 SEM device.

3. Results and Discussion

The theoretical density is 7.99 g/cm³ and experimentally measured density is 7.11 g/cm³, relative density is 88.98% and porosity is 11.02% for unrolled (control) samples, given in Table 2. Hardness of hot rolled samples are given in Table 3 depending on rolling ratio and rolling temperature, given Table 3.

Table 2: Theoretical and experimental density of control sample

<table>
<thead>
<tr>
<th>Theoretical density (gr/cm³)</th>
<th>Experimental density (gr/cm³)</th>
<th>Relative density (%)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.99</td>
<td>7.11</td>
<td>88.98</td>
<td>11.02</td>
</tr>
</tbody>
</table>

Table 3: Hardness values of the samples (HB)

<table>
<thead>
<tr>
<th>Hardness of unrolled sample (HB)</th>
<th>Rolling ratio (%)</th>
<th>Rolling Temperature (°C)</th>
<th>Hardness Brinell (HB)</th>
</tr>
</thead>
<tbody>
<tr>
<td>41</td>
<td>10</td>
<td>600</td>
<td>57</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>700</td>
<td>57</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>800</td>
<td>57</td>
</tr>
<tr>
<td></td>
<td>40</td>
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</table>

Hardness values shown in Figure 5 depending on rolling temperature and in Figure 6 depending on rolling ratio. Hardness is minimum, 41 HB, for unrolled samples, maximum for all rolling temperature 66 HB, minimum for 10% rolling ratio 57 HB. For all samples increasing rolling ratio results in higher hardness. It is seen that there is no significant difference in hardness between the samples rolled at 600°C, 700°C and 800°C for same rolling ratio. The highest hardness values of Cu - B4C obtained at a rolling temperature at 700°C with 40% rolling ratio is 66.3 HB, at 800°C with a roll rate 40% it is 66HB and at 600°C with a 40% roll rate it is 66.2HB. This is due to the deformation hardening caused by the hot rolling process [12]. Minimum hardness of hot rolled samples is 57 HB for 10% rolling ratio and all rolling temperature. Additionally, while increasing in rolling ratio and temperature result in higher hardness there are no significant difference depending on rolling temperature for same rolling ratio. It is also seen that in Fig.5, rolling ratio has strong effect on hardness, especially over 10% rolling ratio.

Figure 5: Rolling temperature’s effect on hardness

Figure 6: Rolling ratio’s effect on hardness

Transverse Rupture Strength (TRS) of the samples given in Table 4, depending on rolling ratio and rolling temperature.

Table 4: TRS values of the samples depending on rolling temperature and rolling ratio

<table>
<thead>
<tr>
<th>TRS of control sample (N/mm²)</th>
<th>Rolling Ratio (%)</th>
<th>Rolling temperature (°C)</th>
<th>TRS (N/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>308</td>
<td>10</td>
<td>600</td>
<td>313</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>700</td>
<td>333</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>800</td>
<td>336</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td></td>
<td>312</td>
</tr>
</tbody>
</table>

Maximum TRS values measured in 10% rolling ratio for all rolling temperature between 331 - 336 N/mm2, nearly same for all rolling temperature. For 600 °C rolling temperature TRS values decreased depending on increasing rolling ratio but higher than control samples. For 700 °C and 800 °C rolling temperature, TRS values reduce for 20% and 30% rolling ratio but it reaches at 40% rolling ratio that maximum TRS values, obtained in 10% rolling ratio (Figure 7 and Figure 8).
**Figure 7:** Rolling temperature’s effect on TRS

**Figure 8:** Rolling temperature’s effect on TRS

**Figure 9:** Fracture surfaces a) Unrolled sample b) Rolled 20% rolling ratio at 800 °C (2500x)

**Figure 10:** SEM images of control sample’s fracture surface (500x)

**Microstructure SEM - EDS analysis**

The SEM micrographs for the samples unrolled and rolled 20% rolling ratio at 800 °C, shown in fig.9a - b, Cu particles seen as light - colored regions, while the black angle shapes indicated B4C particles. The B4C observed uniformly distributed on the compost side of the copper matrix composite, and some pores observed in the compounds [18].

Figure.10 shows fracture surface of unrolled samples after three point bending test, 500x. It is seen that B4C particles clearly distributed in the matrix and there is no segregation in any particular region. However, a porosity of B4C particles was observed [19]. In fig.10 dimples can be observed in non - hot - rolled samples. Brittle fracture in big size B₄C particles occurred, Fig.10).
The small grain size causes B4C to be placed in the gaps in the matrix, thus the reinforcing element is homogeneously distributed in the matrix. The fracture shape of the specimen is brittle. Brittle fracture occurs by inducing a notch effect in boron carbide grains. However, some cracking effects observed on the fracture surfaces of the hot - rolled specimens due to the applied rolling pressure causing the forced motion of the dislocations (fig.11) Large, deep voids and small dimples motifs appear on the fractured surface of each of the hot - rolled composite specimens [13]. The fracture surfaces of both hot - rolled composite specimens analyzed by SEM micrographs and EDS after the (TRS) test (fig.11 a-b). From the SEM and EDS analyses, it is observed that the B4C particles are embedded in the matrix structure. The B4C particles cracked without breaking off from the matrix [22]. The remaining B4C particles on the fractured surface after the (TRS) test are evidence of the good interfacial bonding between the matrix and reinforcement particles. Thus, it can be assumed that the other side of the cracked B4C particles retained the other part of the fractured surface. These embedded B4C particles act as a crack stopper and prevent the rapid progression of cracks through the matrix structure. Thus, the deformation of the composites is limited [23].

EDS analysis (Fig.12) shows the presence of boron (16.84 wt. %), carbon (22.20 wt. %), copper (58.53 wt. %), and oxygen (2.42 wt. %) elements in the structure. This proves the presence of boron carbide in the composite structure. In the SEM image, dark areas indicate B4C, and the remaining gray areas indicate Cu matrix. Upon examination of the other samples, it is seen that B4C and C peaks form, and there are increases in the intensities of these peaks with increasing reinforcement rate. No new phase forms in the structure. It is also understood from EDS analysis that oxide forms, even partially, depending on the increasing temperature on the surface. There were small amounts of oxygen elements in the copper and composite samples fig.12 This probably resulted from the oxidation of the matrix during sintering or specimen preparation [1] [24]. Similar results also obtained from EDS analysis of hot rolled samples.

4. Conclusion

The major conclusions resulting from the work presented in this paper can be listed as follows:

- EDS analyses of composites show that the main components of Cu–B4C composites are copper and B4C, and a small amount of oxide was found on the free surface of B4C particles, especially for composites rolled at 800 °C.
- The hardness of composites increased as the rolling ratio increased at the same rolling temperature.
- It is found that the highest hardness value is 66.3 HBN for 40% rolling ratio and 700°C rolling temperature. For rolling ratio of 10% at all rolling temperature (600°C, 700°C, 800°C) the hardness value is the lowest, and the hardness values are very close at 57 HBN. It is important to note that the hardness values of all samples at different rolling ratio and rolling temperatures are close to each other. The hardness increases as the rolling ratio increases, until the highest hardness values are obtained at a rolling ratio of 40%.
- It is considered that the most important reason for the breakup of B4C reinforcing material is that composite specimens relatively had a porous structure, and these
particles, having a hard construct, caused cavities on the surface of specimens.

- The particles of B4C reinforcing material that initially had sharp edges were partly broken up, spread and smeared over the copper matrix, but this was relatively reduced as the rolling temperature was increased.
- Added B4C reinforcements help in strengthening of the composite by inducing dislocation strengthening mechanisms by obstructing dislocation mobilities at interfaces.
- Hot rolling at 10% can be used for increasing hardness of Cu+B4C composites while TRS values stay nearly steady.
- 10% and 40% rolling ratio have significant effect on increasing of TRS values and hardness.
- Disadvantage of cold pressing can be evaluated by hot rolling.

References


