Production of the Nano-structured composite Brass by Mechanochemical resuscitation of the Copper Oxide

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Abstract: In this research mechanochemical resuscitation of the copper oxide by the Zinc in order to make Nano-structured composite Brass and enhanced solubility limit on the Zinc lattice has been survived. Ball milling on a mixture of the Copper oxide and the Zinc powder to produce the alloy Brass with 35 and 50 atomic percent of the Zink was conducted. XRD results indicated that ball milling a mixture of the Zinc and the Copper oxide with molar ratio 1 to 1 for about 45 minutes can lead to fully reclamation of the Copper oxide and ball milling with Zink molar ratio for 5 hours can lead to make the alloy Brass. The Zinc oxide particles as a reinforcing phase are dispensed with same distribution. By increasing atomic percentage of the Copper to 65, lattice parameter of the alloy Brass has fallen. It represents dissolution of zinc on the Brass lattice α+ β and makes a super-saturated solution of the Brass from proportions of the zinc and the copper.


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Keywords: Mechanochemical resuscitation, the Copper oxide, the Nano-structured composite Brass, super-saturated solution of the Brass.

1. Introduction
The processes of mechanochemical are new methods of materials synthesis which are utilized to produce Nano crystalline materials [1]. In this method reduction of oxide, in ambient temperature and by imposed mechanical energy will be possible [2, 3]. The mechanism of process thus under the occurred collisions between balls and powder grains and wall, in ball shape mill existed powder grains break and cold weld connected and new surfaces continuously are exposed of contact to the factor of resuscitator and resuscitation will be performed. And also based on these collisions, the produced product will contain little crystal structure. With this method, chemical restoration of metal oxides with regenerative agent is appropriate [4]. For the first time mechanochemical resuscitation of metal oxide in the presence of strong regenerative agents was confirmed by the Schaffer and McCormick, they could resuscitate copper oxide (CuO) by using calcium. [5] This method is called Mechanical solid state reduction (MSSR). The metal Sulfide and Chloride could resuscitate in this way and be converted to pure metal [4]. Also so far resuscitation of Copper oxide (CuO) by Ni, Al, Fe, Si, Mn, Ti has been examined [6-7].

2. Materials and Methods of test
Raw materials which are used in this research include copper oxide powder (99% purity, Merck Inc.) and zinc powder (Bakhlys 95% >, Inc. Merck), both with an average particle size of μm45.
In the first part of the test, raw material with stoichiometric ratios based on the following two overall reaction were mixed together to reach the required time for copper oxide and also determines the production of Nano-structured alloy Brass.

\[ \text{CuO}+\text{Zn}\rightarrow\text{ZnO}+\text{Cu} \]
\[ \text{CuO}+2\text{Zn}\rightarrow\text{ZnO}+\text{CuZn} \]

In the second part, in order to increase the solubility limit of copper in combined alloy Brass with Zn-65Cu as shown in Figure 1 is located in the area of two-phase β +α was prepared by Mechanochemical method and its results were compared with the results of Zn-50Cu. Condition of ball milling were put as the pervious experiment.
Mechanical alloying by a planetary ball mill at room temperature and under argon atmosphere rpm600 speed was carried out. Used pellets were made of abrasion-resistant steel and their diameter was mm20. The experiments were carried out with ratio of ball to powder 20:1 and in each experiment 6gr of the original powder were used.

In order to identify the formed phases during mechanical alloying and determination of grain size, X-ray diffraction analysis was used. Testing with Philips machine, Model PW1800, Voltage KV40 and flow of electron mA 40 was done. In all experiments CuKα radiation with a wavelength of 541/1 Å was used. In all experiments, XRD the step size 05/0 and the time step was considered 1 second.

Powder grains size and morphology were investigated by using Scanning Electron Microscope (SEM) VEGA TESCAN model. The average size of grain based on XRD results and using Scherrer equation is calculated.

\[ d = \frac{0.9\lambda}{\beta \cos \theta} \]

In this regard, d is the average size of grains, λ is the X-ray wavelength (1.5405), β is the peak width at half maximum of height and θ is the Bragg angle.

3. Results and Discussion

In order to evaluate the required time for the resuscitation of a mixture the Zinc and the Copper oxide with an equal molar ratio were milled at different times. X-ray diffraction analyses related to these samples are shown in Figure 2. As can be observed in this figure, after 30 minutes of ball milling no reaction based on Copper oxide resuscitation has been done by the Zink. With the passage of 45 minutes of ball milling operations, resuscitation of Copper oxide is carried out completely by the Zink and Copper oxide is converted to pure copper and Zinc is converted to Zink oxide.

The reaction at period of less than 15 minutes indicates that the combustion reaction is carried out.

As the figure 2 shows increasing the ball milling time up to 3 hours, no changes other than intensity reduction of peaks and overspread of them can be seen, its reason can be also reduce the size of the crystals and the strain in the crystal lattice of Copper and Zinc oxide. By using Scherrer equation Copper oxide grains size before restoration and the grain size of Copper metal were determined after resuscitation and is obtained around 25 nanometers (Figure 3).
that during the first time of ball milling cold welding mechanism dominates on fracture mechanism, therefore the grains size is increased. After 30 minutes, due to rising energy imposed on powder grains, imperfections and dislocation of density are increased and fracture process is dominated and the grains sizes are reduced that results in newly formed surfaces of Copper oxide grains are set in the further

Figure 3: grains size of Copper oxide and Copper metal at various times before and after resuscitation based on Scherer equation

In order to evaluate Brass production sample No.2 (a mixture of Zn and CuO in the ratio of 2 to 1 at different times were milled and its results were examined by XRD analysis that is shown in Figure 4 XRD results of milled samples at different times (2) It is observed that over 1 hour of ball milling CuZn solid solution has taken shape however, the first peak that is related to the alloy Brass is appear after 5 hours of ball milling and up to 15 hours; some changes except grains shrink and increased peak width cannot be accessed. Figure 5, EDS image provided by scanning electron microscope indicates the sample which is milled for 10 hours, and quantitative results has been shown in Table 1.

Table 1: Atomic percentage of analyzed grains

<table>
<thead>
<tr>
<th>Element</th>
<th>At %</th>
</tr>
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<tbody>
<tr>
<td>O</td>
<td>35.19</td>
</tr>
<tr>
<td>Cu</td>
<td>21.26</td>
</tr>
<tr>
<td>Zn</td>
<td>43.55</td>
</tr>
</tbody>
</table>

Beside the Brass phase the Zinc oxide grains also exist. And we observe the peak of oxygen. Because of the non-conductive oxide phase on the surface of sample covered by gold which unnamed peak in EDS image related to this cover. From SEM image we can noticed that the produced grains have not arrange and special form. By using Scherer formula size of the grain in different times of ball willing was determined.

Figure 6: The process of reducing size of the grain Brass with the ball milling time

4. Increase the solubility limit of Copper in the alloy Brass
In order to survey the increase of solubility limit of Copper in the α + β Brass lattice, composition of
0.65Cu-Zn as a form of equilibrium is located in α + β zone of phase diagram was prepared by mechanochemical method and its XRD results were compared with the results of Zn-50Cu sample in Figure 7. It is perceived that with enhance of copper in the brass lattice the relevant peaks at this phase of Zn-0.65Cu sample have been transferred to higher angles. In order to survey examined changes more accurately due to rising amount of copper existing in the Nano-structured alloy Brass, parameter of Brass lattice based on angle of peak which is related to the page (110) was calculated (Figure 8).

As it is observed in this figure by increasing percentage of the Copper to 65 atomic percent, lattice parameters of the Brass has reduced. With increasing the Zink, lattice parameters of the Brass has increased and its result is a super-saturated solution of the Brass from proportions of the Zinc and the Copper.

![Figure 7: position of peak (110) in two the Brass](image)

![Figure 8: Changes of lattice parameter to the variations of atomic percent of Copper on the basis of peak (110)](image)

2. Ball milling takes 5 hours to produce the alloy brass and its grains size will be reduced to as small as 14 nanometers after 15 hours' time.

3. With increase of Copper percentage in the alloy Brass, the relevant peaks at this phase will be transferred to higher angles and the lattice parameter will also be decreased.

**References**


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