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Production of nano leaded brass alloy by oxide materials

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Abstract: Mechanochemical process was applied for brass alloy production using chemical grade oxide powders. Alpha brass was produced when CuO, ZnO and PbO were milled under argon atmosphere in the presence of graphite (as reducing agent). Concurrent reduction of zinc, copper and lead oxides took place in the argon atmosphere, whereas the reactions of the reduction did not proceed well in the closed chamber that filled with air. These reactions were promoted by milling time up to 100 hours. The amount of reduction tends to decrease for longer times.

Introduction

Mechanical alloying (MA) is a powder processing technique used for both manufacturing and research. Powder particles are subjected to competing actions of cold-welding and fracture by high-energy ball milling ,which changes their microstructure and chemical composition. Chemical composition of final particles corresponds to the average of the original constituents.
Suryanarayana [1] suggested that MA can be used for alloy production at low temperatures. He mentioned a system of Ni and Cr powders milled until magnetic response was not resolvable in the solid state ,which means that a fine dispersion of one component in the other was achieved. El-Eskandarany work [2] is a good example of this dispersion effect production of very homogenous dispersion of aluminium oxide in aluminium matrix .Indeed, this is the basis for using MA for production of oxide-dispersion-strengthened super alloys, which is MA's main industrial application. MA has produced intimate mixtures of ductile components, such as homogenous mixtures of copper and lead, which are impossible to produce by conventional solidification techniques [3].
Milling time is the most important parameter in mechanical alloying process. Normally, after a while, a balance is achieved between fracturing and cold welding of powder particles. Necessary milling time depends on the type of mill used, intensity of milling ,ball-to-powder ratio and temperature of milling and it may be different for each combination of the above parameters and powder systems. However, it should be noticed that the level of contamination increases and some undesirable phases form, if the powder is milled for longer times than required [3].
Milling atmosphere has also the essential role in powder contamination control. Therefore, powders are usually milled in containers that are either evacuated or filled with an inert gas such as argon or helium. Highly pure argon is the most common gas to prevent oxidation and/or contamination of the powder [4, 5].

In this study, formation of leaded brass by mechanochemical method is investigated using metal oxides and graphite as starting materials. The effects of milling time and atmosphere on this process are evaluated.

Experimental

Pure powders were mixed to give average final composition of 28 wt % Zn, 2 wt %Pb, and balance copper. The initial composition is mentioned in table 1. The copper oxide was purchased from Panrac Company. The zinc oxide was purchased same, with a purity of greater than 99.95%. The lead oxide was with a purity of greater than 99% and the graphite was purchased from Merk Company. Providing the samples are completely reduced, the average final composition would be 28 wt % Zn, 2 wt % Pb and balance copper. In each test the proposed weights of initial powder mixture and balls were supplied in the vessel and the lid was closed. Experiments were carried out for the planned times of 20, 50, 100, 150, 200, 250 and 300 hours. After each experiment the milling was stopped, the lid was opened and sampling was performed by taking 3 grams of powder from the vessel, together with the proportional amount of steel balls to retain the BPR. Then the lid was closed and the next step was started.

Table 1- Weight percent of initial components

Component	Weight Percent (%)
C	12.7
CuO	57.8
ZnO	24.8
PbO	4.7

Alloying was carried out in an Atrittor mixer/mill (figure 1). Stainless steel balls (8 and 10 mm diameters) were used in a cylindrical stainless steel vessel (ID = 75 mm, H = 145 mm). The vessel atmosphere was kept unchanged by means of an O-ring seal. Three set of experiments were carried out in ambient temperature. The first test was performed in air and the two others in argon atmosphere. The Sample weight in first and second tests was 60 grams with ball-to-powder ratio (BPR) 20:1.

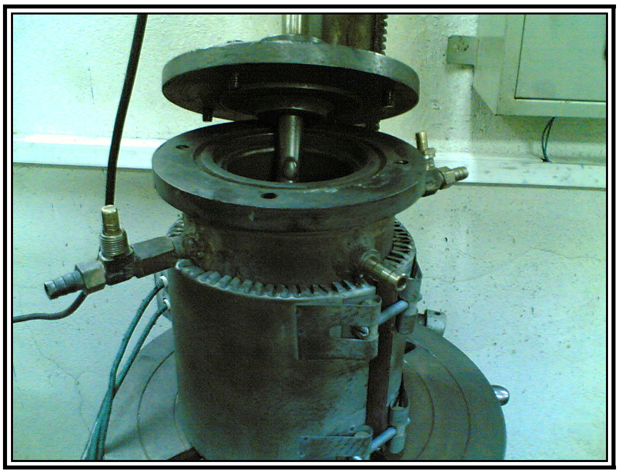

Figure 1- Used attritor mill

In argon experiments, the vessel was rinsed with pure argon (99.99%) for 15 minutes initially and then the inlet and outlet valves were closed during the run. During the MA practice, the vessel was opened at different intervals to take an approximately 3 gr sample for analysis with proportional amounts of balls to retain the BPR. For the practices in argon, the vessel was rinsed with argon for 15 minutes after each sampling, in order to preserve the inert atmosphere.

X-ray diffraction patterns of samples were recorded by Philips PW1140 using copper kα radiation with nickel filter 2θ was scanned from 20 to 80 degrees for all samples with the same intensity scale. Results were traced to find out the influence of time and atmosphere. Particle sizes of samples were investigated by scanning electron microscope (SEM) TESCAN VEGAII XMU model. Some points on the samples were analyzed by a RONTEC EDS analyzer.

Results and Discussions

X-ray diffraction patterns of first test samples are compared with the initial mixture of CuO, ZnO, PbO and graphite in figure 2. It is obvious that lead oxide and graphite are undetectable in all samples. This behavior can be explained by the low amount of lead oxide in the initial mixture and the high negative Gibbs free energy change of its reduction. Because of its low amount, the reduced lead cannot be detected in X-ray patterns.

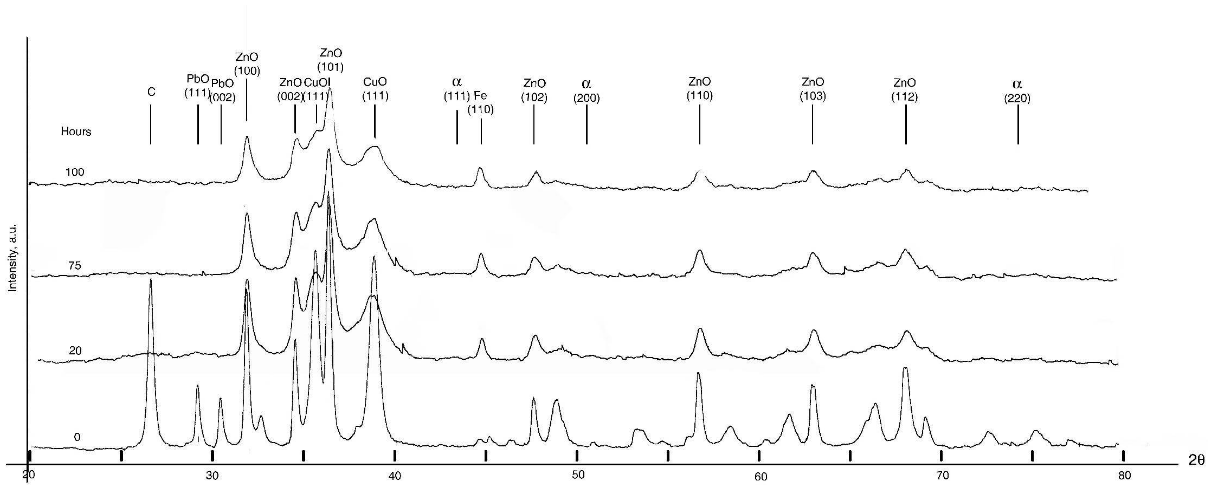

Figure 2- Effect parameter time of mechanical alloying in atmosphere, ambient temperature, composition (12.7%C- 57.8% Cu- 24.8% ZnO- 4.7% PbO) BPR= (20/1)

Decrease of graphite peak intensity can be explained by its consumption in reduction reactions and by its dissolution in other phases. Peaks of copper oxide and zinc oxide gradually become shorter and wider. Size of grains in powder particles reduces due to the severe deformation caused by balls strokes. The X-ray patterns of smaller grains usually show shorter and wider peak.
Formation of brass cannot be detected even after 100 hours milling which can be justified by the oxidizing atmosphere in the vessel. In order to decrease the oxygen potential, the next test was carried out under argon atmosphere. Results are shown in figure 3.

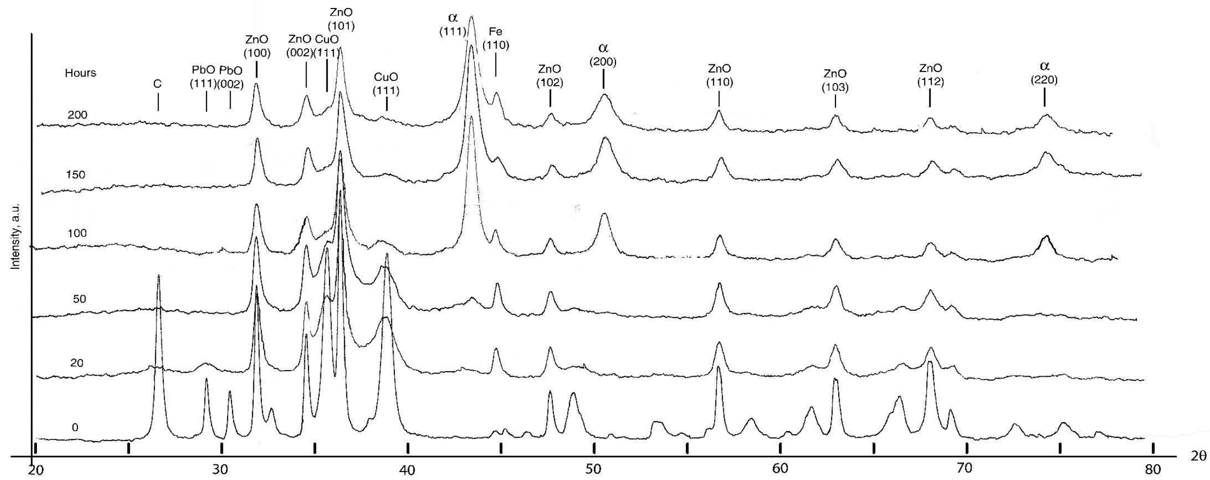

Figure 3- Effect parameter time of mechanical alloying in argon atmosphere, ambient temperature, composition (12.7%C- 57.8% Cu- 24.8% ZnO- 4.7% PbO) BPR= (20/1)

After 20 hours, intensity of oxides peaks decrease and peaks of brass become visible. Intensity of brass peaks reaches a maximum after 100 hours. These peaks become smaller in longer times although the peaks of oxides continue to shorten. This effect can be explained by the formation of micro or nano-size grains .

XRD patterns of samples which are milled for 100 hours at different atmospheres are compared in figure 4. While the solid solution of copper and zinc is formed in argon atmosphere, no metal can be detected in air. Although the reduction of zinc oxide is not possible in experimental conditions of tests, its reduction is favored by dissolution of zinc in the newly reduced copper.

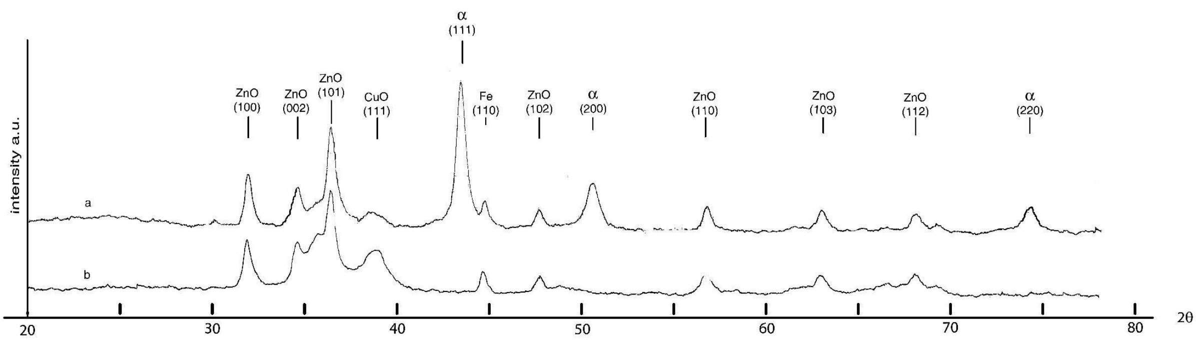

Figure 4- Effect parameter atmosphere, A. Argon, B. Air, in ambient temperature, milling time 100 hours, composition (12.7%C- 57.8% Cu- 24.8% ZnO- 4.7% PbO) BPR= (20/1)

Scanning electron micrograph of milled sample for 20 h, at BPR=20/1, Ar atmosphere and room temperature can be seen in fig. 5. The smallest particle size of this sample as shown figure 5 is about 50 nanometers.

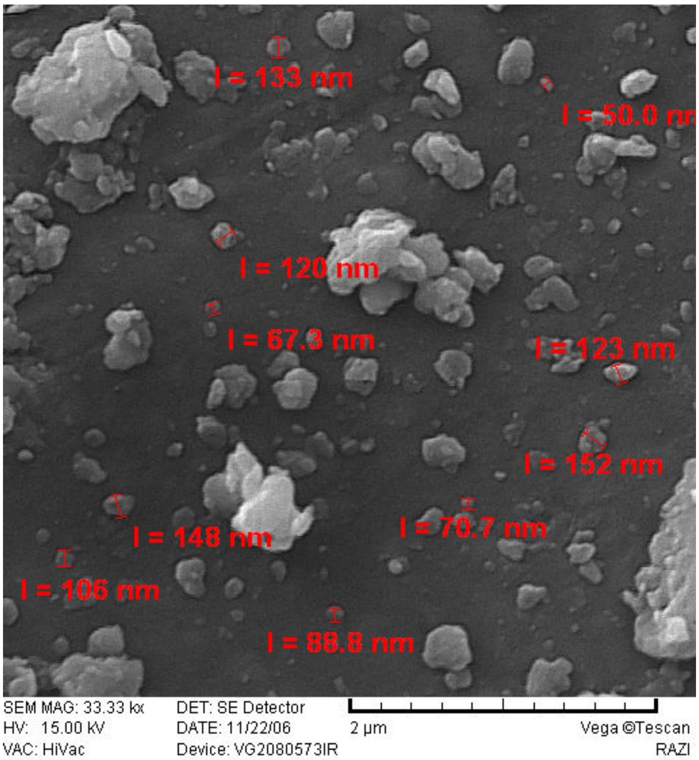

Figure 5- Scanning electron micrograph of milled sample for 20 h, at BPR=20/1, Ar atmosphere and room temperature

Conclusion

1. At room temperature and for the BPR equal to 20/1, no reduction was occurred in air even after 100 hours milling.
2. At same condition with argon atmosphere, initial peak of product can be detected after 20 hours milling. The maximum intensity of brass peak is at 100 h. With increase of time after 100 hours, intensity of brass peak is decreased because of decline of grains size.
3. Formation of copper after milling helps zinc oxide reduction by dissolution of the product.
4. The peak of zinc oxide decreases with time but doesn't eliminate until the end.

References:

[1] C. Suryanarayana, "Mechanical alloying and milling ", Progress in Materials Science, Vol 46, (2001)
[2] M. S. El-Eskandarany, “Mechanical Alloying for Fabrication of Advanced Engineering Materials”, William Andrew Publishing, (2001).
[3] Y. Chank, S. Berger, B. Z. Weiss, Broolc-Levinson, “Solid State Amorphization by Mechanical Alloying- an Atomistic Model”, Acta Matter, vol. 42, No. 11, (1994) p. 3679-3685
[4] B. T. McDermott and C. C. Koch Dept. "Preparation of Beta Brass by Mechanical Alloying of Elemental Copper and Zinc". Scripta Metallurgica. Vol. 20, (1986) p. 669-672
[5] C. Suryanarayana, "Mechanical Alloying and Milling", 1887, Department of Metallurgical and Materials Engineering, Colorado School of Mines , Golden , CO, 80401, USA
[6] M. Enaiati, Z. Sadeghian, “Mechanical alloying principles & applications”, Iranian metallurgical engineering society Journal, Vol 23, (2005) p. 14-19
[7] M. Enaiati, “Mechanical alloying of Ni-Nb powder”, 4’Th annual metallurgical

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References

[1] C. Suryanarayana, "Mechanical alloying and milling ", Progress in Materials Science, Vol 46, (2001)
doi:10.1016/S0079-6425(99)00010-9

[2] M. S. El-Eskandarany, “Mechanical Alloying for Fabrication of Advanced Engineering Materials”, William Andrew Publishing, (2001).

[3] Y. Chank, S. Berger, B. Z. Weiss, Broolc-Levinson, “Solid State Amorphization by Mechanical Alloying- an Atomistic Model”, Acta Matter, vol. 42, No. 11, (1994) p. 3679-3685
doi:10.1016/0956-7151(94)90433-2

[4] B. T. McDermott and C. C. Koch Dept. "Preparation of Beta Brass by Mechanical Alloying of Elemental Copper and Zinc". Scripta Metallurgica. Vol. 20, (1986) p. 669-672
doi:10.1016/0036-9748(86)90487-4

[5] C. Suryanarayana, "Mechanical Alloying and Milling", 1887, Department of Metallurgical and Materials Engineering, Colorado School of Mines, Golden, CO, 80401, USA

[6] M. Enaiati, Z. Sadeghian, “Mechanical alloying principles & applications”, Iranian metallurgical engineering society Journal, Vol 23, (2005) p. 14-19

[7] M. Enaiati, “Mechanical alloying of Ni-Nb powder”, 4’Th annual metallurgical