Nanocrystalline Zn and Its Alloy Analyzed by Miniaturized Disk Bent Test (MDBT)

ZHU Xin-kun[1], LI Cai-ju[1], ZHAO Kun-yu[1], C. C. Koch[2]
(1. Faculty of Materials and Metallurgical Engineering, Kunming University of Science and Technology, Kunming 650093, China;
2. Dept of Materials and Engineering, North Carolina State University, Raleigh, NC 27695- 7907, USA)

Abstract Bulk nanocrystalline pure Zn and Zn- Al alloy can be produced by ball milling. The mechanical properties of those materials are analyzed by tensile test and miniaturized disk bent test (MDBT). The results show that Zn- 0. 6% (wt) Al ball milled for 3~ 5 hours has very good ductility. The pure Zn ball milled for 3 hours at room temperature has better ductility than pure Zn cryomilling (CM) ball milled for 3 hours at liquid nitrogen and then at room temperature (BM) for 6 hours.

Key words: nanocrystalline; ball milling; Zn; namo-sized Zine
CLC: TG146. 13 **Document code**: A **Article ID**: 1007- 855X(2005)06- 0018- 03

用 MDBT分析纳米 Zn及其合金

朱心昆[1], 李才巨[1], 赵昆渝[1], C. C. Koch[2]
(1. 昆明理工大学 材料与冶金工程学院, 云南 昆明 650093;
2. Dept of Materials and Engineering, North Carolina State University, Raleigh, NC 27695- 7907, USA)

摘要: 应用高能球磨机械合金化方法制备纳米 Zn及 Zn- Al合金体材料, 采用拉伸实验和 MDBT实验对这些材料的样品的机械性能进行测量分析. 结果表明: 球磨 3~ 5 h获得的 Zn- 0. 6% Al含金具有很好的塑性. 室温下球磨 3 h所得的纯 Zn比液氮温度下球磨 3 h加室温球磨 6 h所得的纯 Zn具有更好的塑性.
关键词: 纳米晶; 球磨; 锌; 纳米锌

0 Introduction

The miniaturized disk bent test (MDBT) was used to study the mechanical properties of metal [1~7]. The majority of MDBT techniques were qualitative analysis in the early times. Recently this technique has been improved and used for quantitative analysis for different kinds of studies of metal properties, including Young's modulus, fracture toughness. In most cases regarding with the nanocrystalline materials, the ductility of the materials is the major concern when using MDBT. The MBDT has developed over the past 20 years, which is used to analyze the mechanical properties of materials using small samples. This paper describes the bulk nanocrystalline Zn and its alloy was produced by ball milled and their ductile property was analyzed using MDBT and tensile test.

1 Experiment

The raw powders were prepared from Zn powder with purity 99. 9% and average particle size of 50 μm and

Received date: 2004- 10- 08. **Foundation item**: Supported by National Natural Science Foundation of China (Grant No. 50264002).

Biography of the first author: ZHU Xin-kun (1959~), male, Professor and Supervisor of Ph. D student of Kunming University of Science and Technology. **E- mail**: xk_zhu@ hotmail. com

Al powder with purity 99.97% and a particle size of 75 μm, respectively. The powders were putted into a tool steel vial in a glove box with a purified argon atmosphere. Martensitic stainless steel balls (6.4 mm and 7.9 mm diameter) were used with a ball-to-powder ratio of 10∶1. The ball milling was carried out in a Spex 8 000 shaker mill at liquid nitrogen and room temperatures. After Zn element powders were milled at liquid nitrogen temperature, these powder milled were again milled at room temperature. Zn balls (Maximum 10 mm in diameter) were formed after ball milling at room temperature and over 6 hours. These balls were pressed into disks at room temperature and vacuum under a uniaxial pressure of around 300 MPa. MDBT was measured on Universal Testing Machine. The MDBT samples have thickness of 250~600 μm. The samples were tested in the ring-on ring (ROR) mode MDBT. Details about this technique were reported in [4]. The displacement rate for the actual MDBT was chosen to be $(2.1\sim2.4)\times10^{-3}$ mm/s, which corresponds to the stain rate of approximately $(1\sim4)\times10^{-3}$/s. Microhardness of samples were tested by microhardness instrument.

2 Results and Discussion

The researches indicated that there are five regions of the typical load-displacement curve for ductile materials in the MDBT. They are bulk plate response in elastic regime. Governed primarily by Young's modulus (region 1). Departure from linearity is due to through thickness and subsequent radial propagation of the yield surface from the region of contact with the punch (region 2). Transition from bending to membrane stress regime occurs in most regions of the plate (region 3). Membrane stress regime is dominant in most regions of the plate. These stresses eventually lead to fracture near the plate center on the bottom face (region 4). Fracture occurs somewhere in the region. Load drop is due to: ① through thickness thinning near punch tip ② Fracture with subsequent through thickness and circumferential crack propagation (region 5).

Fig. 1 shows that the curve of samples in MDBT is also typical load-displacement curve, which include above five regions of typical ductile materials. The characters of curves have not affected by the time of ball milling. All displacement distances of three curves are about 1 mm. This indicates their ductility is the same. The samples with different maximum forces are due to their different thick. Fig. 2 shows the results of MDBT in pure Zn. The displacement distance of this curve is about 0.9 mm. By comparing with the Fig. 1, the ductility of Zn has been increased after a little Al was added to Zn to form Zn-Al alloy.

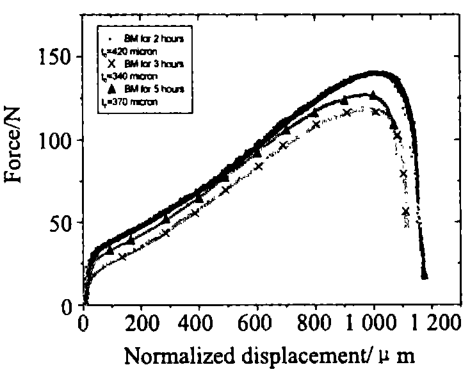

Fig.1 Force and displacement curves of Zn+0.6% Al in MDBT

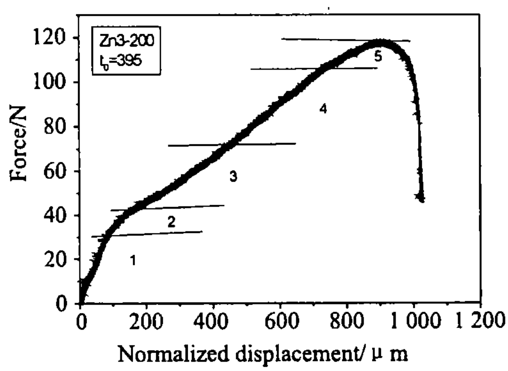

Fig.2 Typical load-displacement curve for pure Zn ball milled for 3 h at room temperature, then annealed for 1 h at 200℃

Fig. 3 shows that there were only 3 regions in load-displacement curve which has not character curve of ductile materials in MDBT. Its maximum displacement distance is about 0.63 mm. This exhibit the sample has poor ductile properties.

The same samples as Fig. 1, Fig. 2 and Fig. 3 were tested using tensile test to analyze their mechanical properties in detail (Fig. 4). The annealing process can increase the ductility of Zn ball milling to 150% elongation.

The elongation of the sample which is the same as Fig. 3 is only 65%. The longest elongation of Zn-0.6% Al ball milled for 3 hours at room temperature is 274%.

Fig. 5 shows that although Zn-Al alloy has lower hardness than pure Zn in different ball milling time, Zn-0.6% Al and Pure Zn have almost alike fracture stress. Al not only improves Zn ductility but also decreases its micro-hardness and keeps its fracture stress on the same level as pure.

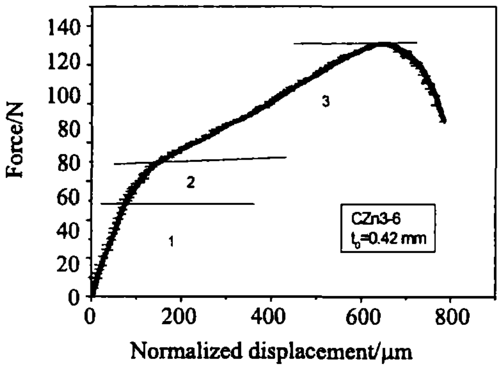

Fig.3 Typical load-displacement curve for pure Zn ball Zn milled for 3 liquid nitrogen(CM) and 6 h room temperature(BM)

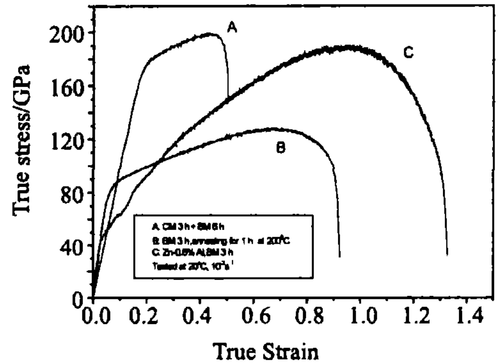

Fig.4 Stress and strain curve of Zn, Zn alloy (A,B is pure Zn, C is a alloy of Zn-0.6% Al)

3 Conclusion

Bulk nanocrystalline pure Zn, Zn-Al alloy was synthesized using ball milling and press. The mechanical properties of materials were analyzed by MDBT. The Zn-0.6% (wt) Al ball milled at room temperature for 3~5 hours has best ductility. The pure Zn ball milled for 3 hours at room temperature has greater displacement distance than pure Zn CM for 3 hours and BM for 6 hours, and the former has also better ductility than the latter.

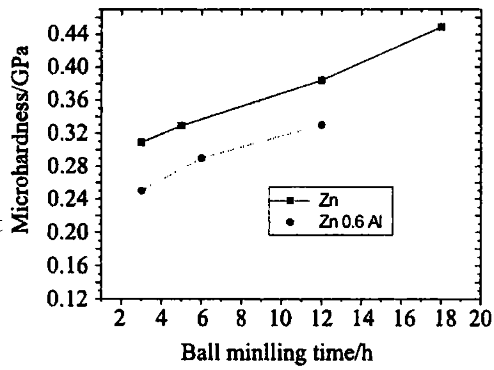

Fig.5 Relation of microhardness and ball milling

Acknowledgment

Mr. ZHU made research work in North Carolina State University as visiting scholar. He would like to thank C. C. Wu Cultural and education Foundation Fund for financial supporting, acknowledges the support from National Science Foundation of China under grant number 50264002.

References

[1] Manahan M P, Argon A S, Harling O K. The development of a miniaturized disk bend test for the determination of post-irradiation mechanical-properties[J]. J Nucl Mater, 1982, 103-104: 1545~1556

[2] Lucas G E. Metall [J]. Trans. 1990, 21(A): 105~112

[3] Meyers D E, Chen F C, Zhang J, et al [J]. J. Test Eval, 1993, 21: 263~271.

[4] Hoffman M, Birringer R. Quantitative measurements of Young's modulus using the miniaturized disk-bend test[J]. Materials Science and Engineering A, 1995, 202 (1-2): 18~25.

[5] Chen Felix C, Ardell Alan J [J]. Mat Res Innoat, 2000, 3: 250~262

[6] Zhang Jin in, Ardell Alan J [J]. J Am. Ceram. Soc, 1993, 76(5): 1340~1344

[7] Manaham M P, Browning A E, Argon A S, et al. The Use of Small-Scale Speciment for Testing Irradiated Materials[M]. ASTM STP 888, W. R. Corwin and G. E. Lucas, Eds, American Society for Testing and Materials, Philadelphia, 1986: 17~49.