第28卷,第7期
2008年7月

光谱学与光谱分析
Spectroscopy and Spectral Analysis

Vol. 28,No. 7,pp1665-1669
July, 2008

硼酸镁晶须增强镁基复合材料的谱学表征与性能研究

金培鹏[1,2],丁雨田[1],许广济[1],刘　岩[2]

1. 兰州理工大学甘肃省有色金属新材料省部共建国家重点实验室,甘肃 兰州　730050
2. 青海大学镁技术工程研究所,青海 西宁　810016

摘　要　用X射线衍射谱分析了$Mg_2B_2O_5w$/AZ91D镁基复合材料中的物相。研究了固溶处理和时效处理以及固溶处理加人工时效处理对复合材料组织演变的影响及组织与显微硬度之间的关系。结果表明,经过固溶处理后,共晶相的分解使复合材料的硬度值明显下降。时效处理使得复合材料的硬度逐渐增加并在时效处理16 h后出现时效峰值201 HV。然而随着时效时间的进一步增加,显微硬度值降低。经固溶处理24 h,基体中β相基本溶解,形成过饱和的固溶体,接着时效处理8 h,β相以弥散形式析出,从而使得复合材料的显微硬度值提高30%;而固溶处理24 h及时效处理24 h后,β析出相由细小片状的连续析出相向粗大的层片状非连续析出相过渡,使得复合材料的显微硬度下降到183 HV。

关键词　镁基复合材料;X射线衍射谱;热处理;显微组织;硬度
中图分类号:O657.3　　**文献标识码**:A　　**文章编号**:1000-0593(2008)07-1665-05

引　言

增强金属材料常用晶须是碳化硅、硼酸铝晶须[1-11]等。碳化硅晶须价格昂贵,用于航天等领域。硼酸铝晶须价格低廉,性能优越,但在增强合金时,会与合金中的镁等元素发生界面反应[12-15],这又限制了其应用。

硼酸镁晶须性能较优越,价格更为低廉,在硼酸镁晶须增强镁基复合材料中尚未发现界面反应现象,展示出诱人的应用前景。

关于硼酸镁晶须增强镁基复合材料的研究极少。热处理对合金及其复合材料组织与性能的影响一般都用显微组织观察和显微硬度的变化进行研究[16,17]。显微组织观察结合X射线衍射光谱研究金属基复合材料在各种热处理制度下的报道很少见到,周延怀等曾对GeO_2-SiO_2纳米复合材料经X射线衍射分析,观察到随热处理温度的增高,材料结构从无序到有序的转变过程[18]。最近的文献主要集中在研究金属基复合材料热处理后复合材料内所产生的内应力方面[19,20]。然而,由于用显微组织观察组织演变难于定量确定组织中相的溶解和析出,而X射线衍射光谱可很好地分析在不同热处理制度处理后复合材料中物相的变化。

本文采用固溶处理和时效处理,用X射线衍射光谱分析了热处理过程中复合材料中物相的变化,研究了热处理过程对复合材料的金相结构及性能的影响。

1　实验部分

1.1　实验材料

实验材料是体积分数为30%的$Mg_2B_2O_5w$/AZ91D镁基复合材料。

1.2　实验过程

在MEF-3大型光学显微镜上观察金相组织变化。采用HX-1000TM硬度仪测定不同热处理状态的复合材料和基体合金的维氏硬度。

热处理在YFFG49/13G-YC型电阻炉上进行,炉内温差±5 ℃。热处理工艺参数如表1。采用D8 ADVANCE型X射线衍射仪对试样进行X射线衍射分析。

Table 1　Heat-treatment parameters for the $Mg_2B_2O_5w$/AZ91 composites

固溶处理(T4)	时效处理(T5)	固溶+时效处理(T6)
415 ℃8,24 h热水冷却	200 ℃ 8,12,16,20,24 h空冷	415 ℃8,24 h热水冷却+200 ℃8,24空冷

收稿日期:2007-06-06,**修订日期**:2007-09-09
基金项目:中国科学院2005年度"西部之光"人才培养计划项目资助

作者简介:金培鹏,1961年生,青海大学镁技术工程研究所教授　　e-mail:jinpeipeng@mail2.lut.cn

2 结果和讨论

2.1 X射线衍射图谱和固溶处理对复合材料显微组织和硬度的影响

图1所示是铸态下复合材料的X射线衍射图谱。可看出，铸态下复合材料中有β $Mg_{17}Al_{12}$相的峰。固溶处理过程中，β相溶入α Mg相中形成固溶体。如图2(a)所示，经固溶处理8 h，可以看出β相已大部分溶入α Mg相中生成固溶体，还有少部分未溶入的以共晶相存在。随着固溶时间的延长，β相的溶入更加充分。如图2(b)所示经过415 ℃固溶处理24 h后，β相完全溶入α Mg中，对复合材料固溶24 h后的X射线衍射图谱见图3。可以看出，已没有β相的存在，复合材料由α Mg相、$Mg_2B_2O_5$ w以及表面的MgO构成，固溶效果明显。

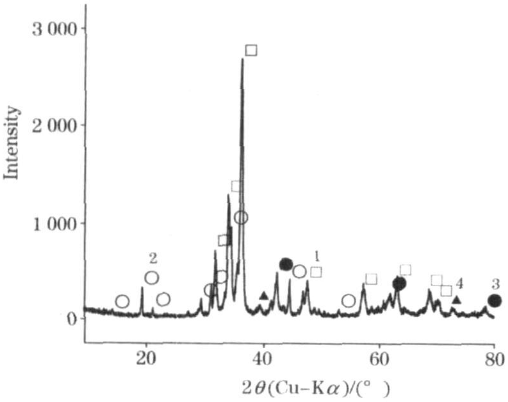

Fig. 1 X-ray diffraction pattern of the as-cast $Mg_2B_2O_5$ w/AZ91D composite

1: Mg; 2: $Mg_2B_2O_5$; 3: MgO; 4: $Al_{12}Mg_{17}$

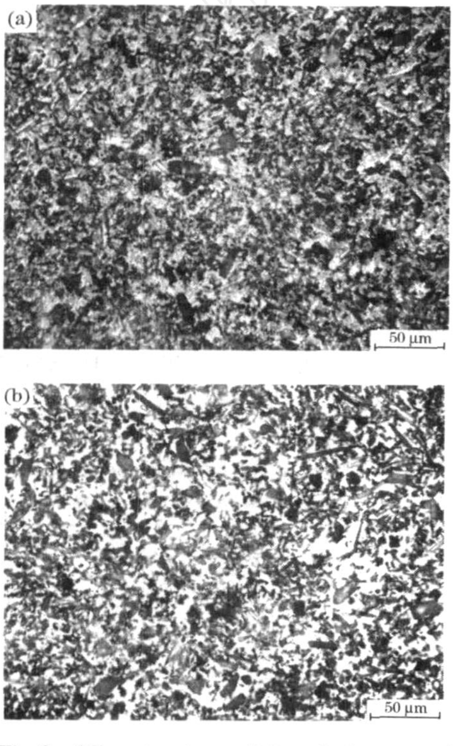

Fig. 2 Microstructures of the solution treated $Mg_2B_2O_5$ w/AZ91D composites

(a): After solution treated for 8 h at 415 ℃;

(b): After solution treated for 24 h at 415 ℃

图4所示为不同固溶时间复合材料硬度的变化。从图中看出，随固溶时间的延长，合金的硬度逐渐下降。这是由于经过固溶处理后，基体镁合金中β相分解溶入α Mg相中形成过饱和固溶体，这种组织硬度较低，导致了复合材料硬度下降。

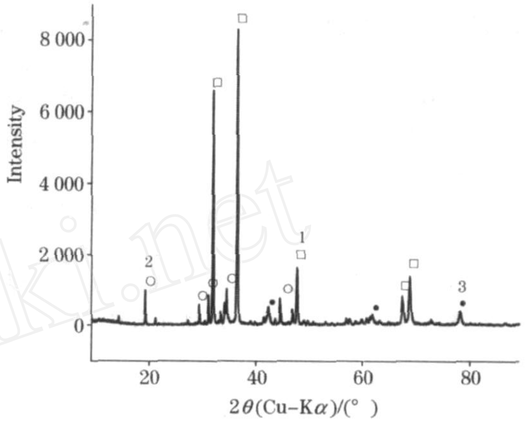

Fig. 3 X-ray diffraction pattern of $Mg_2B_2O_5$ w/AZ91D composite after solution treated for 24 h at 415 ℃

1: Mg; 2: $Mg_2B_2O_5$; 3: MgO

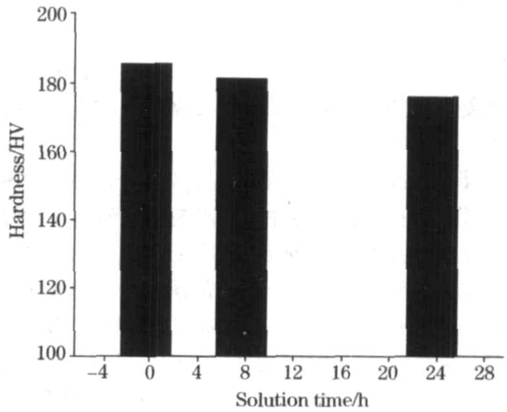

Fig. 4 Vickers hardness of the $Mg_2B_2O_5$ w/AZ91D composite after solution treated at 415 ℃ for different time periods

2.2 时效处理对复合材料显微组织和硬度的影响

图5为不同时效处理后复合材料的金相组织，显示出了不同时间时效处理后复合材料金相组织的变化。

图6是不同时效时间后复合材料硬度变化曲线。可看出，随着时效时间的增加，复合材料硬度值逐渐增加，说明时效强化效果明显，时效处理16 h出现时效峰值。进一步时效处理，硬度开始降低。原因是时效过程中，由于β相的析

出，析出相起到了明显的弥散强化效果。随着时效时间的进一步增加，析出相形貌发生很大变化。由颗粒状向大片块状过渡，这种集聚降低了复合材料力学性能。

2.3 固溶+时效热处理对复合材料显微组织和硬度的影响

图 7 为不同固溶+时效处理后复合材料金相组织。由图 7(a)中看出，固溶和时效时间短，β相的析出较少。固溶 8 h

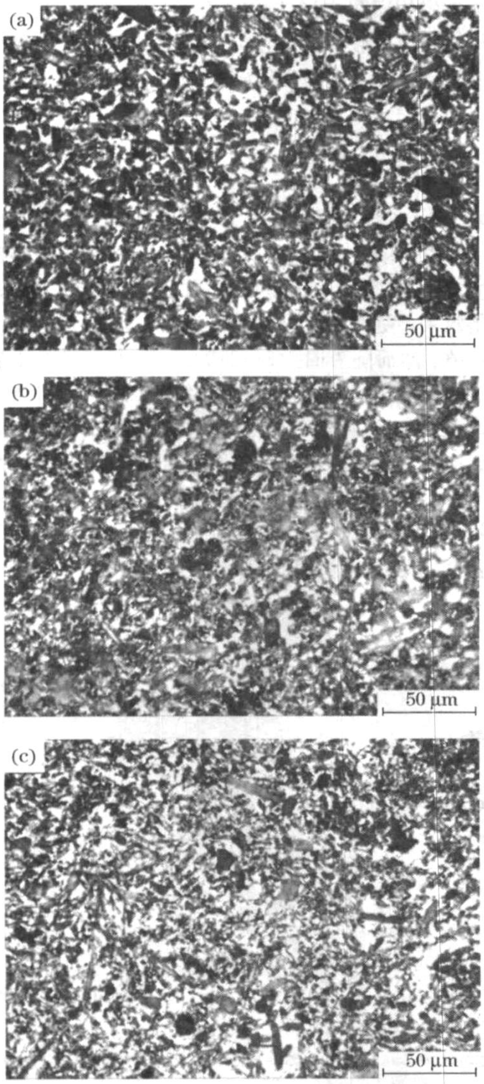

Fig. 5 Microstructures of $Mg_2B_2O_5$ w/AZ91D composites after isothermal aged at 200 ℃ for different time periods

(a): After aged for 8h; (b): After aged for 16 h; (c): After aged for 24 h

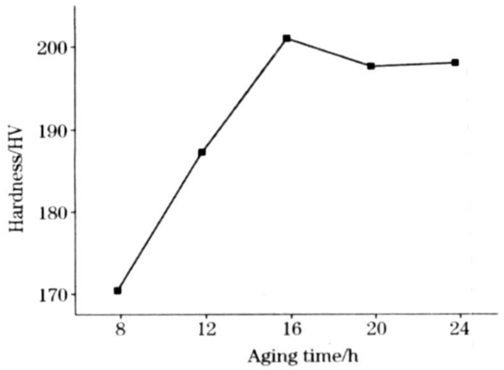

Fig. 6 Curve of vickers hardness for $Mg_2B_2O_5$ w/AZ91D composites after isothermal aged at 200 ℃ for different time periods

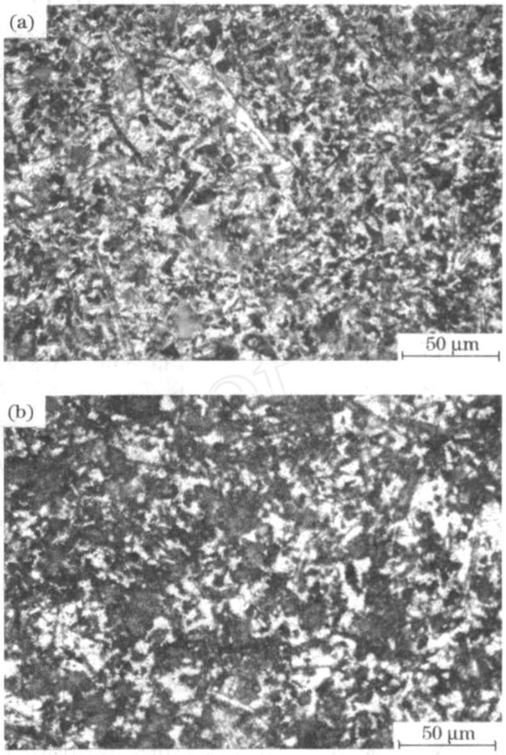

Fig. 7 Microstructures of $Mg_2B_2O_5$ w/AZ91D composites after different solution and subsequent aging treated process

(a): After solution treated for 8 h at 415 ℃ and subsequent aging treated at 200 ℃ for 8h;

(b): After solution treated for 24 h at 415 ℃ and subsequent aging treated at 200 ℃ for 24 h

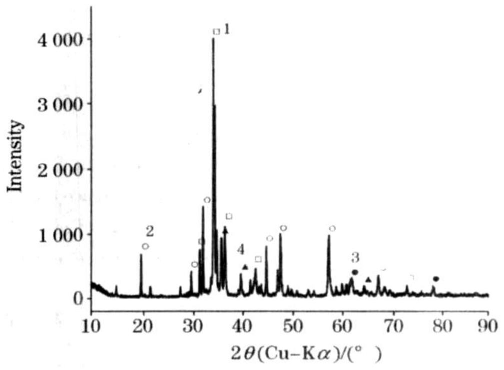

Fig. 8 X-ray diffraction pattern of $Mg_2B_2O_5$ w/AZ91D composite after solution treated for 24 h at 415 ℃ and subsequent aged for 24 h at 200 ℃

1: Mg; 2: $Mg_2B_2O_5$; 3: MgO; 4: $Al_{12}Mg_{17}$

+时效 8 h 处理过程中，β相从非连续析出开始，固溶 24 h+时效 24 h 后，形成大块连续的分布。对固溶 24 h+时效 24 h 的试样进行 X 射线衍射分析见图 8。可以看出，复合材料中重新出现β相的峰。

表 2 所示为不同热处理工艺后复合材料的硬度值。可以看出时效 8h 处理下，固溶处理时间长的硬度值大。原因是 24 h 固溶处理后，β相的分解溶入更加充分，使得基体组织中的过饱和程度明显大于 8 h 固溶处理的过饱和程度，在时效处理过程中，由于过饱和程度大，β相析出速率快且更加均匀，起到了明显的弥散强化效果；时效处理 8 h，复合材料的硬度大幅度提高，而时效处理 24h 后，复合材料的硬度又大幅度下降。主要原因是随着时效时间的延长，β相由细小片状的连续析出相向粗大层片状的非连续析出相过渡，这种大块的聚集使弥散度较低，对合金的强化作用较小，降低了复合材料的力学性能，使硬度值降低。

Table 2　Vickers hardness of $Mg_2B_2O_5w$/AZ91D composites after different heat treated process

工艺参数	固溶 8 h， 时效 8 h	固溶 24 h， 时效 8 h	固溶 8 h， 时效 24 h	固溶 24 h， 时效 24 h
硬度(HV)	230.2	240.8	190.2	183

3　结　论

研究发现，经过 415 ℃固溶处理 24 h 后，β相完全溶入αMg 相中，固溶热处理 $Mg_2B_2O_5w$/AZ91D 复合材料的硬度下降。而时效处理由于β相的析出强化了复合材料，起到了弥散强化效果，使得复合材料的硬度逐渐增加并在时效 16 h 后出现峰值。然而随着时效时间增加，析出相形貌由颗粒状向大片块状过渡，这种集聚使硬度降低。固溶处理 24 h，接着时效处理 8 h，基体组织中的高饱和度使得β析出相的数量多且更加均匀，起到了弥散强化效果，使得复合材料的显微硬度值提高 30%；而固溶处理 24 h，接着时效处理 24 h 后，β相由细小片状连续析出相向粗大层片状非连续析出相过渡，这种大块的聚集，弥散度较低，使得复合材料的硬度下降。

参　考　文　献

[1] Sasaki G, Adachi J, Choi Y B, et al. PRICMS: The Fifth Pacific RIM International Conference on Advanced Materials and Processing, PTS1-5, 2005, 475-479: 921.

[2] Zheng M Y, Wu K, Liang H C, et al. Materials Lett., 2002, 57(3): 558.

[3] Yoshida M, Takeuchi S, Pan J, et al. Advanced Composite Materials, 2001, 10(2, 3): 255.

[4] Sasaki G, Yoshida M, Yanagisawa O, et al. Materials Science Forun, 2003, 419-422: 777.

[5] Zhang X, Zhang D, Wu R, et al. Scripta Materialia. 1997, 37(11): 1631.

[6] Chang S Y, Tezuka H, Kamio A. Materals Trausaction, JIM. 1997, 38(1): 18.

[7] Zhang X N, Geng L, Wang G S. J. Material Process Technology, 2006, 176(1-3): 146.

[8] Hua Yunfeng, Zhang Litong, Cheng Laifei, et al. Materials Science and Engineering A, Structural Material Properties Microstruct Processing, 2006, 428(1-2): 346.

[9] Borrego Alberto, Fernández Ricardo, Cristina M D, et al. Composites Sci. Tech., 2002, 62(6): 731.

[10] Hu Lianxi, Wang Erde. Materials Science and Engineering A, Structural Material Properties Microstruct Processing, 1999, 278(1-2): 267.

[11] Keesam Shin, Sunghak Lee, Sung-Joon Kim, et al. Adv. Performance Materials, 1998, 5(4): 307.

[12] Yao L J, Sasaki G, Fukunaga H. Materials Science and Engineering A, Structural Material Properties Microstruct Processing, 1997, 225: 59.

[13] Zheng M Y, Wu K, Liang M, et al. Materials Science and Engineering A, Structural Material Properties Microstruct Processing, 2004, 372: 66.

[14] BI Gang, WANG Hao-wei, WU Ren-jie, et al. Tran. Nonferrous Met. Soc. China, 1999, 9(4): 785.

[15] Pan J, Sasaki G, Yao L J, et al. Mater. Science and Technology, 1999, 15: 1044.

[16] CHEN Ti-jun, MA Ying, HAO Yuan, et al. Trans. Nonferrous Met. Soc. China, 2001, 11(1): 98.

[17] Hassan S B, Aigbodion V S. Materials Science and Engineering A, Structural Material Properties Microstruct Processing, 2007, 454-455: 342.

[18] ZHOU Yan-huai, FENG Yu-ying, LU Hai-yan(周延怀，冯玉英，陆海彦). Spectroscopy and Spectral Analysis(光谱学与光谱分析), 2000, 20(1): 23.

[19] Funaki K, Takago S, Fujii K, et al. Sci. and Technology. of Advanced Materials, 2005, 6(8): 902.

[20] Fernández P, Bruno G, González-Doncel G. Composite Sci. Tech., 2006, 66:1738.

Spectroscopic Characterization and Properties of $Mg_2B_2O_5$ w/ AZ91D Magnesium Composites

JIN Pei-peng[1,2], DING Yu-tian[1], XU Guang-ji[1], LIU Yan[2]

1. State Key Laboratory of Gansu Advanced Non-ferrous Metal Materials, Lanzhou University of Technology, Lanzhou 730050, China
2. Institute of Magnesium, Qinghai University, Xining 810016, China

Abstract Magnesium borate whisker ($Mg_2B_2O_5$ w) reinforced Mg matrix composite was fabricated by vacuum-gas pressure infiltration process. The $Mg_2B_2O_5$ w preforms forming process was determined. The $Mg_2B_2O_5$ whiskers were fabricated into a preform by wet forming method without any binder. The Vacuum-Gas Pressure Infiltration process and parameters are also developed. The micrographs revealed reasonably uniform distribution and random orientation of the whiskers in the as-cast $Mg_2B_2O_5$ w/ AZ91D composite and the composites were without pores defect. The phases were analyzed by XRD patterns for the as-received whiskers, the whiskers sintered at 1 000 ℃ for 3 h and the as-cast composite, respectively. Then, the microstructure evolution of the composite was investigated when the composite was heat-treated. Meanwhile, the relationships between microstructure and micro-hardness of the alloy heat-treated were also studied. The heat-treatment condition were solution at 415 ℃ for 8 and 24 h, respectively, aging treatment at 200 ℃ for 8, 12, 16, 20 and 24 h, respectively and solution at 415 ℃ for 8 or 24 h and subsequent aging treated for 8, 12, 16, 20, 24 h, respectively. The phases were analyzed by XRD patterns for the composites after different heat treated process. The results of XRD patterns were shown to be in good agreement with the microstructures evolution of the composites. The results showed that the micro-hardness of the solution treated composites is decreased due to resolution of eutectic phase, whereas the micro-hardness of the aged composites was increased gradually and the peak hardness is reached to 201 HV in the composite aged for 16 hours. Solution treatment at 415 ℃ for 24 h, the β-$Mg_{17}Al_{12}$ phase is dissolved in the α-Mg phase to form oversaturated solid-solution in the composite and then the diffusive β phase precipitates after subsequent aging treatment at 200 ℃ for 8 h; hence the micro-hardness of the composite was increased 30%. However, as the aging time increased to 24 h, the hardness of the composite was reduced to 183 HV because the β-$Mg_{17}Al_{12}$ phase precipitate changed from continuous fine platelets to discontinuous coarse platelets. It was concluded that the process of solution at 415 ℃ for 24 h and subsequent aging treatment at 200 ℃ for 8 h was the best process for the composite.

Keywords Magnesium based composite; XRD; Heat treatment; Microstructure; Vickers hardness

(Received Jun. 6, 2007; accepted Sep. 9, 2007)