Available online at www.sciencedirect.com



Cite this article as: Rare Metal Materials and Engineering, 2009, 38(8): 1325–1327.

ARTICLE

# Phase Transformation and Magnetic Properties of Dy<sub>0.5</sub>Fe<sub>60</sub>Pt<sub>39.5</sub> Alloy

Cheng Gang, Gu Zhengfei, Zhou Huaiying

Guilin University of Electronic Technology, Guilin 541004, China

**Abstract:** The effects of the annealing time on the microstructure and magnetic properties of  $Dy_{0.5}Fe_{60}Pt_{39.5}$  were investigated. X-ray diffraction and *M-T* data indicated that the disordered face-centered-cubic phase (soft phase) is not truly disordered phase due to an increase in the degree of short-range order with increasing of annealing time. Both the coercivity and remanence ratio of the FePt based alloy are as a function of the annealing time. The optimum heat treatment condition for the alloy is at annealing temperature of 600 °C for 5 h. These results of magnetic properties can be explained on the basis of phase transformation and microstructure of the alloy.

Key words: Fe-Pt based alloys; Curie temperature; grain size; magnetic properties

The FePt-based alloys have received significant attention owing to their excellent application potential such as data storage and dental applications<sup>[1-3]</sup>. Generally, the crystal structure of the FePt alloy which are melted and then quenched to room temperature is face-centered-cubic (fcc)  $\gamma$ -FePt disordered phase and shows soft magnetic behavior<sup>[4]</sup>. This disordered fcc *γ*-FePt phase can be transformed into hard magnetic face-centered-tetragonal (fct)  $\gamma_1$ -FePt ordered phase after annealing at appropriate temperature. Many efforts have been done to enhance the permanent magnetic properties by phase transformation and microstructure control<sup>[5-7]</sup>. Q. F. Xiao et al found that a larger number of the nearest-neighbor Fe atoms to an Fe atom leads to a lower Curie temperature in  $Fe_{59.75}Pt_{39.5}Nb_{0.75}$  alloy, and the Curie temperature of the disorder phase and order phase for FePt is about 300 and 400 °C, respectively<sup>[8]</sup>. Y. K. Takahashi et al found that the ordering temperature upon the addition of 4 at% Cu to the FePt film was decreased approximately by a factor of 200 °C<sup>[9]</sup>. Hao Zeng et al investigated the effect of the annealing temperature on magnetic properties of the FePt film and found that a peak value of coercivity appeared at annealing temperature 600 °C<sup>[10]</sup>. In the present work we report the effect of annealing time on the phase transformation and magnetic properties of Dy<sub>0.5</sub>Fe<sub>60</sub>Pt<sub>39.5</sub> based alloy.

#### **1** Experimental

The ingots of the composition Dy<sub>0.5</sub>Fe<sub>60</sub>Pt<sub>39.5</sub> was prepared by arc-melting of pure metals (the purities of the ingredients were more than 99.9 wt%) under purified argon. They were remelted not less than four times to ensure good homogeneity. The as-arc-melted alloy was cast in the form of a thin cylinder and then was pressed into plates of about 0.4 mm thickness, 3 mm width and 10 mm length for XRD test. The samples were sealed in quartz tubes pre-evacuated and refilled with some purified argon, homogenized at 1150 °C for 3 h, and then quenched in ice water. Subsequently, the as-quenched samples were annealed at 600 °C for different time. The analysis of crystal structure and the phase composition was carried out by XRD with Cu Ka radiation. The magnetic properties were measured with a PPMS-9T at room temperature, and the maximum applied field was 3 T. M-T curves were measured with a vibrating sample magnetometer. The Curie temperature  $T_{\rm C}$  was determined from *M*-*T* curves. The lattice constants of the compound under study can be derived using Gufi5 software to treat the data of XRD with high-purity Si as the internal standard. The mean grain (ordered region) sizes were calculated from the broadening of the X-ray diffraction profiles.

Received date: July 27, 2008

Foundation item: Supported by the National Natural Science Foundation of China (50261002&50661002).

Biography: Cheng Gang, Ph. D., Professor, Department of Electronic Information Materials Science and Engineering, Guilin University of Electronic Technology, Guilin 541004, P. R. China, Tel: 0086-773-5601434, E-mail: cheng59@guet.edu.cn

Copyright © 2009, Northwest Institute for Nonferrous Metal Research. Published by Elsevier BV. All rights reserved.

### 2 Results and Discussion

Fig.1 shows X-ray diffraction patterns of Dy0.5Fe60Pt39.5 alloy heat-treated at 600 °C for different time. The degree of the tetragonal distortion of the ordered fct phase can be easily estimated from the apparent splitting of the (200), (002), (220), (202), (311) and (113) reflections, originating from the (200), (220) and (311) reflections of the original fcc phase. The XRD patterns clearly show the fcc phase, as depicted in Fig.1 for homogenized  $Dy_{0.5}Fe_{60}Pt_{39.5}$  sample. As annealing time increases from 1 to 100 h, the intensity of reflections belonging to the  $\gamma_1$ -FePt phase increases, and the (200), (220) and (311) reflections of the original y-FePt phase split to the (200), (002), (220), (202), (311) and (113) reflections of the original  $\gamma_1$ -FePt phase. According to the XRD data in Fig.1, the amount of fct phase increases with increase of annealing time but the y-FePt phase does not really disappear even though the annealing time reaches 20 h (see Fig.2). Fig.2 shows the temperature dependence of the magnetization of Dy<sub>0.5</sub>Fe<sub>60</sub>Pt<sub>39.5</sub> alloy annealed at 600 °C for different time. The Curie temperature of Dy<sub>0.5</sub>Fe<sub>60</sub>Pt<sub>39.5</sub> depends strongly on the number of the nearest-neighbor Fe atoms to a Fe atom, and there are six nearest-neighbor Fe atoms around a Fe atom in the ideally disordered and stoichiometric fcc phase. This is in contrast to the ordered fct phase where for the equiatomic composition each Fe atom has an average of four nearest-nighbor Fe a toms. The reference literature[11] stated that the Curie temperature in  $Pt_{1-x}Fe_x$  alloys decreases with increasing of x in the concentration range considered. This suggests that a lower number of Fe nearest neighbors to a Fe atom makes  $T_{\rm C}$  increase. There are two Curie temperatures in the M-T curves of the samples by annealing treatment as depicted in Fig.2. The lower  $T_{\rm C}$  value and higher  $T_{\rm C}$  value should correspond to the fcc phase and fct phase, respectively. The Curie temperature of fcc phase increases as the annealing time increases. It is suggested that soft-magnetic phase in the sample is not a truly soft-magnetic phase, instead, cubic phase with degree of short-range order. Due to the fact that the sample annealed for



Fig.1 XRD patterns of Dy<sub>0.5</sub>Fe<sub>60</sub>Pt<sub>39.5</sub> alloy obtained by quenching after homogenization at 1150 °C and annealing at 600 °C for different time



Fig.2 Temperature dependence of the magnetization  $of \ Dy_{0.5}Fe_{60}Pt_{39.5} \ alloy$ 

1 h has two Curie temperatures, this means that the ordered fct phase has started to form in the sample. But the degree of long-range order is still fairly low. The different degrees of atomic order can exist not only in the fct phase but also in the fcc phase. The degree of short-range order increases with increasing of annealing time. Theinterm ediate cubic phase with short-range order remains in the process for the phase transformation from fcc phase to fct phase. As seen in Fig.2, if the annealing time reaches 20 h, a considerable amount of the fcc phase has still remained, despite the fact that the fct phase has become the main phase.

Fig.3 shows the dependence of the remanence ratio and average grain size of the ordered phase on the annealing time. It can be seen that the grain size of the fct phase increases rapidly during the initial annealing stage (1-20 h). The remanence ratio as a function of the annealing time first increases rapidly as annealing time increases from 1 to 5 h, and then decreases with further increases of annealing time. A peak value about 0.69 of  $M_r/M_s$  appears at annealing time 5 h for average grain size D=22 nm. It can be explained as follows: according to the model of Kneller and Hawig<sup>[12]</sup>, the remanence ratio increases as the grain size decreases because of exchange coupling between



Fig.3 Remanence ratio and grain size of the ordered phase of  $Dy_{0.5}Fe_{60}Pt_{39.5}$  alloy as a function of the annealing time, at annealing temperature of 600 °C

soft magnetic and hard magnetic phase. On the other hand, the soft magnetic phase is not truly soft magnetic phase because of increase in the degree of order in the fcc phase. The two factors of the grain size and the degree of order in fcc phase are counterbalanced, resulting in remanence ratio peak value of 0.69 at annealing time for 5 h and grain size for 22 nm.

Fig.4 illustrates the effect of annealing time on the magnetic properties of  $Dy_{0.5}Fe_{60}Pt_{39.5}$  alloy. The coercivity  $H_c$  of  $Dy_{0.5}Fe_{60}Pt_{39.5}$  alloy increases rapidly from 120 to 260 kA/m and then decreases with increasing of annealing time. When the annealing time is 5 h, the strong exchange interaction occurs between ordered phase and disordered phase (as shown in Fig.3). Exchange coupling between soft magnetic and hard magnetic phases can effectively increase the coercivity of FePt based alloys. Additional, the anisotropy constant  $K_k$  of the hard magnetic phase. However, the anisotropy constant  $K_k$  is closely related to the coercivity  $H_c$ . The relationship between coercivity and anisotropy constant can be expressed as<sup>[12]</sup>:

 $H_{\rm c}=K_{\rm k}/\mu_0 M_{\rm sm}$ 

where  $M_{\rm sm}$  is the saturation magnetization of soft magnetic phase and  $K_{\rm k}$  the anisotropy constant of the hard magnetic phase. On the one hand, the heat treatment can lead to increasing in degree of order of fct phase. On the other hand, the heat treatment also leads to soft magnetic phase being not truly soft magnetic phase. These two factors are counterba-



 $\label{eq:Fig.4} \begin{array}{ll} \mbox{Effect of annealing time on magnetic properties} \\ \mbox{of } Dy_{0.5}Fe_{60}Pt_{39.5} \mbox{ alloy annealed at } 600 \ ^\circ C \end{array}$ 

lanced, resulting in coercivity  $H_c$  peak value of 260 kA/m at annealing time 5 h for average grain size D=22 nm. Maximum  $(BH)_{max}$  value of  $Dy_{0.5}Fe_{60}Pt_{39.5}$  alloy annealed at 600 °C for 5 h could reach about 70 kJ/m<sup>3</sup>.

#### 3 Conclusions

1) The grain size of the ordered phase changed from 6 nm to 37 nm upon annealing at 600  $^{\circ}$ C for different time.

2) The fcc phase is not truly disordered phase due to an increase in the degree of short-range order.

3) When the  $Dy_{0.5}Fe_{60}Pt_{39.5}$  alloy is quenched after homogenization at 1150 °C and then annealed at 600 °C for 5 h, the coercivity, remanence ratio, and  $(BH)_{max}$  could reach a maximum value of approximately 260 kA/m, 0.69, and 70 kJ/m<sup>3</sup>, respectively.

#### References

- Klemmer T J, Shukla N, Liu C et al. Appl Phys Lett[J], 2002, 81: 2220
- 2 Brück E, Xiao Q F, Thang P D *et al. Physica B*[J], 2001, 300: 215
- 3 Klemmer T, Hoydick D, Okumura H et al. Scr Metall Mater[J], 1995, 33: 1793
- 4 Watanabe K. Mater Trans JIM[J], 1991, 32:292
- 5 Liu J P, Luo C P, Liu Y et al. Appl Phys Lett[J], 1998, 72: 483
- 6 Tanaka Y, Kimura N, Hono K et al. J Magn Magn Mater[J], 1997, 170: 289
- 7 Xiao Q F, Brück E, Zhang Z D et al. J Alloys and Compounds[J], 2004, 364: 315
- 8 Xiao Q F, Bruck E, Zhang Z D et al. Physica B[J], 2003, 339: 228
- 9 Takahashi Y K, Ohnuma M, Hono K. J Magn Mater[J], 2002, 246(1-2): 259
- Zeng Hao, Sun Shouheng, Vedantam T S et al. Appl Phys Lett[J], 2002, 80: 2583
- Hansen M. Constitution of Binary Alloys[M]. New York: McGraw-Hill, 1958: 698
- 12 Kneller E F, Hawig R. *IEEE Transactions Magnetics*[J], 1991, 27: 3588

## Dy0.5Fe60Pt39.5合金的相转变和磁性能

成 钢,顾正飞,周怀营 (桂林电子科技大学,广西 桂林 541004)

**摘 要**:研究了退火时间对 Dy<sub>0.5</sub>Fe<sub>60</sub>Pt<sub>39.5</sub> 合金结构和磁性能的影响。通过热磁曲线的测量获得了软磁相和硬磁相的居里温度。实验表明: 由于退火时间的增长,合金中的短程有序度增加,使无序的面心立方相并不是真正的无序相。合金的矫顽力和剩磁比都是退火时间的函 数,最佳的热处理条件是在 600 ℃ 退火 5 h。根据合金相转变和微结构的变化对磁性能的结果进行了解释。 关键词: Fe-Pt 合金;居里温度;晶粒尺寸;磁性能

作者简介:成 钢,男,1959年生,博士,教授,桂林电子科技大学电子信息材料科学与工程系,广西 桂林 541004,电话:0773-5601434, E-mail: cheng59@guet.edu.cn