Granular Nanostructures and Magnetic Properties of FePt-C/FePt-SiO_2 Films

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The $L1_0$ ordered FePt is one of the most promising candidates for heat assisted magnetic recording (HAMR) media due to its high bulk magnetocrystalline anisotropy energy constant K_u of ~7 × 10^7 ergs/cc. For HAMR media, $L1_0$ FePt-X(segregant) thin films must have high coercivity and small, uniform and columnar shape FePt grains. Various segregants, such as C, TiO_2, SiO_2 and etc. [1-3] have been doped to FePt film to obtain desired properties. By doping amorphous SiO_2 and TiO_2 can fabricate the (001) textured FePt films with columnar grains. However, these FePt films exhibited poor perpendicular anisotropy because their phase separation tendency is too weak to isolate FePt grains in the lateral direction[4]. FePt-C granular films realize high K_u and well-isolated FePt grains with small grains. But the doped C easily diffused to the surface at the relatively higher fabrication temperature of FePt films. This resulted in second nucleation and the formation of double layer structure with increased media thickness[5]. In this work, we successfully fabricated columnar structured FePt film with large coercivity by using FePt-C/FePt-SiO_2 bilayer structure. Granular nanostructures and magnetic properties of FePt-C, FePt-SiO_2 and FePt-C/FePt-SiO_2 films have been investigated.

Fig. 1(a) shows an XRD pattern of the FePt-C30*vol*%(4 nm)/FePt-SiO_2 45*vol*%(4 nm) film. The wide background peak between 17 ° and 35 ° corresponds to the amorphous glass substrate. The (001) and (002) peaks of the $L1_0$ FePt structure are clearly observed with a missing (111) peak, indicating the FePt grains are strongly (001) textured. The high degree of chemical ordering of $L1_0$ FePt manifests itself as a large integrated peak intensity ratio $A(FePt_{001})/A(FePt_{002})$=2.2. Fig. 1(b) shows the magnetization curves of the film. Coercivity of the out-of-plane direction is about 28.5 kOe. Fig. 1(c) and (d) show the cross-sectional TEM bright field images of the film. It is evident that only one layer of well-isolated columnar FePt grains which are about 10 nm in diameter and 13 nm in height is epitaxially grown on the MgO intermediate layer. It can be seen that the FePt-SiO_2 layer is successfully grown on the FePt-C layer without forming the second layer. Note that the 8 nm thickness of FePt-C/FePt-SiO_2 is thicker than the critical thickness of FePt-C single-layer formation (4 nm). It means that the FePt-SiO_2 layer suppress the phase separation between FePt and C.

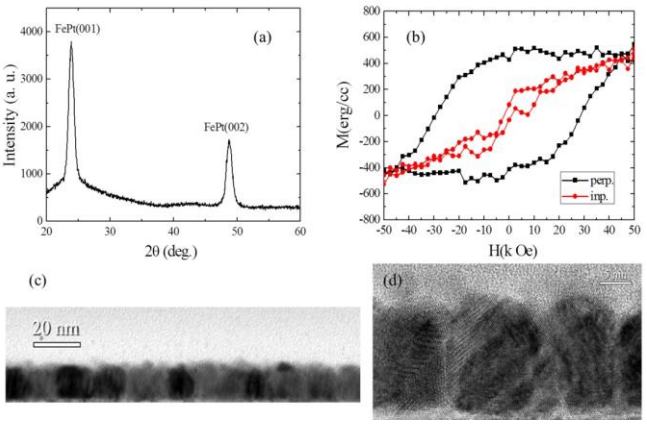

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Fig. 1. (a) XRD pattern, (b) magnetization curves and (c), (d) cross sectional TEM images of FePt-C30*vol*%(4 nm)/FePt-SiO_2 45*vol*%(4 nm) film.

Simulation of $L1_0$ FePt microstructure by using phase field model

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Heat Assisted Magnetic Recording (HAMR) media demands $L1_0$ FePt-X(segregant) thin films should have small and columnar FePt grains with high coercivity. In this work, the influence factors to form the columnar FePt grains were studied by using 3D phase field model based on Koyama's model.[1)]

Fig. 1 shows the 3D microstructure of the FePt-X thin films with increasing the film thickness ($L1_0$ FePt: yellow, $A1$ FePt：red, X: cyan). The simulated volume is 50×50×(t=2-10) nm^3 and using isotropic atomic mobilities. The interfacial energy is 1.82 J/m^2. Fig. 1(a) shows the morphology of the FePt-X thin films when t is 2 nm, and the columnar FePt grains can be seen clearly. Fig. 1(b) shows the FePt-X microstructure when t is 5 nm. The bilayer FePt grains start to form and the interconnected FePt grains increase. Fig. 1 (c-d) shows that the FePt grains are layer by layer or semi-spherical shape when the FePt-X thickness varies from 8 nm to 10 nm.

Fig. 2 shows the variations of 3D microstructure of the FePt-X thin films with the different mobility M_{cz} values and the same mobility M_{cx}=M_{cy}=1.0. The volume is 50×50×10 nm^3. Fig. 2 (a)-(b) shows the number of bilayers of FePt grains reduces when M_{cz} is decreased from 0.5 to 0.1. When M_{cz} continues to decrease to 0.01 and 0.001, the microstructure of the FePt-X thin films almost fully become the columnar shape as shown in Fig. 2(c) and Fig. 2(d). These results clearly demonstrate that selecting the materials with the anisotropic mobility of atom diffusion as a segregant is vital to prepare the columnar microstructure of the FePt-X thin films.

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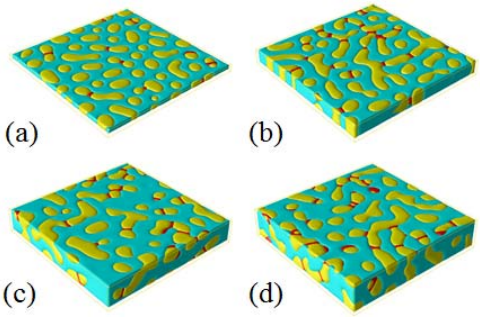

Fig. 1 The variations of the FePt-X thin films microstructure with different film thicknesses: (a) 2 nm; (b) 5 nm; (c) 8 nm; (d) 10 nm.

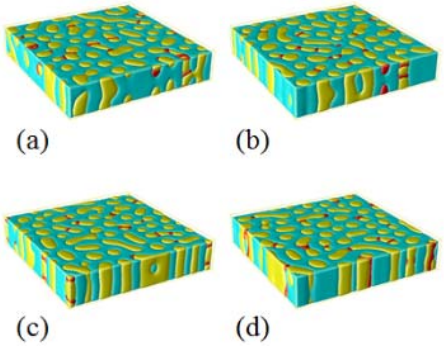

Fig. 2 The FePt-X thin films microstructure with decreasing M_{cz} at t = 10 nm: (a) 0.5; (b) 0.1; (c) 0.01; (d) 0.001.

Effect of amorphous Cr-Ti texture inducing layer on highly (002) textured large grain $Cr_{80}Mn_{20}$ seed layer for $L1_0$ ordered FePt-C granular film

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Introduction Heat-assisted magnetic recording media has attracted much attention for future hard disk drive owing to the high magnetocrystalline anisotropy of $L1_0$ ordered FePt ($K_u \sim 7\times10^7$ erg/cm^3) [1]. In order to improve signal to noise ratio, it is necessary to fabricate the medium in which the grains are magnetically isolated such as FePt-C based granular film. For large perpendicular anisotropy, controlling the *c*-axis (002) orientation of the FePt, corresponding to the magnetic easy axis, toward normal to the film is required. In general, the preferred crystal orientation of the FePt can be adjusted by means of hetero-epitaxial growth from (002) textured MgO underlayer. However, it is reported that the misalignment of the (002) grains normal to the film is caused by angular distribution of the MgO (002) orientation [2, 3]. Origin of the angular distribution is regarded due to the absence of the epitaxy growth of the MgO underlayer, which is directly deposited on an amorphous film. In this study, method to suppress the angular distribution is proposed by introducing a new concept of layered structure.

Concept of layered structure The proposed structure consists of the magnetic layer (ML)/under layer (UL)/seed layer (SL)/ texture-inducing layer with amorphous structure (*a*-TIL) as shown in Fig.1. Developing the SL with having the highly (002) texture-crystalline film, key idea of the concept, can be described when satisfying the high wettability of SL on *a*-TIL. The liquid phase of sputtered atoms of the SL tends to spread out on the *a*-TIL before solidification. Since the crystal terrace, evolved during solidification from liquid phase, possesses the largest surface area on the top surface compared to the other surfaces, the main contribution to the sheet texture evolution results from the top surface. And consequently, the large grain size with highly textured SL can be realized. Typically, the condition of high wettability is defined as Young relation written below $\gamma_{SL} > \gamma_{in}+\gamma_{a\text{-TIL}}$ where γ_{SL} is surface energy of SL, γ_{in} is interfacial energy, and $\gamma_{a\text{-TIL}}$ is surface energy of *a*-TIL. Since there is still lack of information on γ_{in}, expectation for high wettability by using the interfacial energy is difficult. However, it can be rationalized by taking into account the quantitative value between γ_{SL} and $\gamma_{a\text{-TIL}}$. Accordingly, condition favorable for the high wettability can be a high value of $\gamma_{a\text{-TIL}}$ and a lower value γ_{SL}. Thus, we have investigated to enlarge the grain size of SL by changing the quantitative value of $\gamma_{a\text{-TIL}}$.

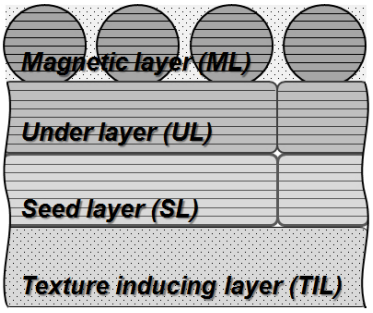

Fig. 1 Concept of layered structure for suppressing the (002) texture distribution of FePt-C medium.

Experimental results CrMn and Cr-Ti were introduced as the SL and *a*-TIL, respectively [5]. In order to enlarge the grain size of SL, two methods were presented. (1) High wettability; changing the compositions of Cr-Ti *a*-TIL owing to the higher γ_{Ti} (2.570 J/m^2) than γ_{Cr} (2.056 J/m^2) [5]. (2) Promoting the adatomic mobility of the SL; elevating the substrate temperature. The film structure used in this study consists of CrMn (30)/$Cr_{100-x}Ti_x$ (20)/$Ni_{60}Ta_{40}$(2)/glass substrate. Substrate temperature was elevated before CrMn deposition. The temperature varied from RT to 600 °C. Ti composition (x) in the $Cr_{100-x}Ti_x$ *a*-TIL varied from 0 to 100 at.%. Figure 2 shows the dependence of the full width of half maximum at CrMn (002) diffraction ($FWHM_{002}$) on the grain diameter (*GD*) of the CrMn SL with various fabrication conditions of Cr-Ti TIL. The $FWHM_{002}$, degree of the angular distribution, was evaluated by rocking curve profile (not shown in this abstract). The GD was estimated by Scherrer equation using CrMn (110) diffraction measured by in-plane XRD. As shown in the figure, the $FWHM_{002}$ decreases from 10.5 deg to 3.4 deg with increase of the *GD* from 11.4 nm to 15 nm. The result indicated that the remarkable progress in reducing the one-third value of the angular distribution was accomplished by increase of 3 nm of *GD*. It is expected that the epitaxy from CrMn, MgO to FePt-C can be improved by introducing Cr-Ti *a*-TIL.

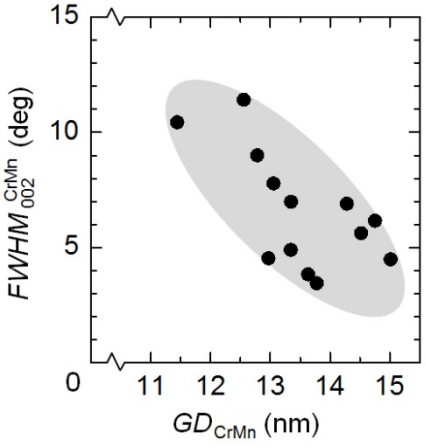

Fig. 2 $FWHM_{002}$ for CrMn seed layer dependence on grain diameter GD_{CrMn} with various fabrication conditions of Cr-Ti TIL.

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Switching field distribution of FePt-C/FePt exchange coupled perpendicular media

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Since the grain size of the recording media has to be reduced, a magnetic material with high K_u must be applied for the concern of thermal stability. However, high K_u media require high writing field in order to write the information onto the media. Due to the physical limitation where the maximum attainable head field is about 1.7 T [1], the writability of such media becomes a challenge. In this work, by increasing FePt soft layer thickness in FePt-C/FePt exchange coupled granular/continuous (CGC) perpendicular media, we successfully reduced the coercivity field from 4.9 to 1.4 T without sacrificing thermal stability. Meanwhile, the switching field distribution (SFD) of the bilayer media also got significant improved. Both of these advantages make FePt-C/FePt CGC media here a potential candidate for extremely high areal density recording media which is writable meanwhile thermal stable.

FePt-C 10 nm/FePt X nm exchange coupled granular/continuous perpendicular bilayer films were DC magnetron sputtered on single-crystalline MgO (001) substrates. Bottom hard layer was deposited by the co-sputtering using Fe, Pt and C targets at a substrate temperature of 600°C under 0.48 Pa Ar while the top relative soft FePt layer was sequenced co-sputtered at a lower substrate temperature of 400°C. The soft FePt layer thickness was varied from 2 to 15nm.

Figure 1 shows the in plane and cross-sectional TEM images of MgO(001)/FePt-C 10nm/FePt Xnm CGC perpendicular bilayer films with different soft FePt capping layer thickness. Fig.1 (a) & (b) illustrate the TEM images of single FePt-C 10nm layer without soft capping layer. One can see that the single FePt-C layer gives a well-isolated nano granular structure with average grain size around 10.2 ± 1.5 nm (Inset of Fig. 1a). For bilayer film with 5 nm capping soft FePt layer, due to the inter-diffusion at FePt-C/ FePt interface, FePt grains grow larger with average grain size around 12 nm and the capping soft FePt growth epitaxially on the top of each individual hard FePt grains forming identical grains. Further increase the layer thickness to 10 or 15nm, one can detect soft FePt gains island on the top of bottom FePt grains, finally forming continuous soft FePt layer which is typically CGC structure. On the other hand, magnetization curves (Fig. 2) of the exchange coupled bilayer with various soft FePt layer thicknesses indicate that the introduced capping soft FePt layer also holds perpendicular magnetic anisotropy (PMA) and can effectively reduce the covercivity field Hc (4.9 to 1.4T) though direct exchange coupling at the FePt-C/ FePt interface. Furthermore, SFD analysis with ΔH (M, ΔM) method [2] shows that the direct exchange coupling at the interface can significantly narrow the SFD (33% to 6%) by increasing soft FePt layer thickness. However, decline of SFD with 15nm capping soft FePt layer can be attributed to the degradation of PMA.

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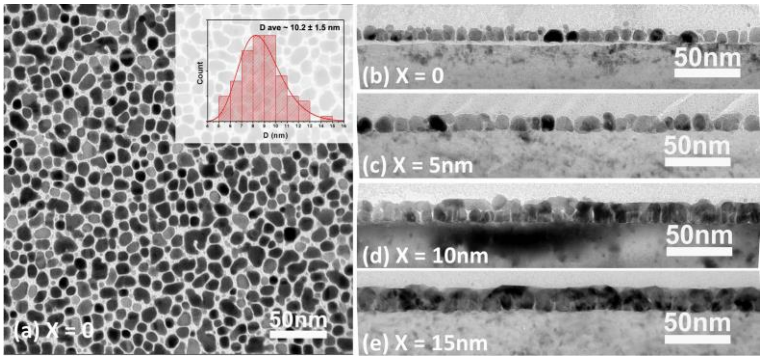

Fig. 1 In plane and cross-sectional TEM images of FePt-C10nm/FePt Xnm exchange coupled media (Inset: FePt grain size distribution)

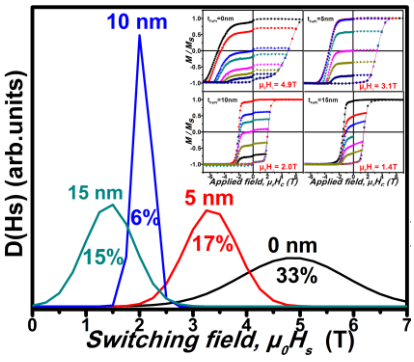

Fig.2　Switching field distribution and corresponding out-of-plane magnetization curves of FePt-C 10 nm/FePt X nm exchange coupled media.

Microstructure and magnetic properties of $L1_0$ ordered FePt-C nanogranular films: Influence of graded structure with different C volume fraction

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$L1_0$ ordered FePt nanogranular thin films are considered as one of the leading candidates for heat assisted magnetic recording media [1] with areal density beyond 1 Tbits/in^2. Considering the feature of its high magnetocrystalline anisotropy, it is possible to fabricate thermally stable FePt particles with size down to 4 nm [2]. Hence, enormous efforts are being made to produce FePt grains with minimum size, high coercivity and columnar growth with the aspect ratio of more than 1.5. However, to realize the nanogranular structure in FePt films, various spacer materials such as C, SiO_2, Al_2O_3, ZrO_2, TiO_2, Cr_2O_3, etc must be used. Although the spacer materials help to reduce the grain size below 7 nm with narrow size distribution, the magnetic properties degrade due to reduction in degree of $L1_0$ ordering and enhancement of misaligned FePt grains.

Therefore, in this study, we present FePt-C granular films deposited as graded layer structure with different C volume fraction by co-sputtering FePt alloy and C targets on single crystalline (001) MgO substrate as a model system. The graded structure was implied to suppress growth of randomly oriented grains on top of FePt granular layer [3] and thickness of FePt-C film was optimized for obtaining a single layered structure with columnar growth. Crystal structure and degree of $L1_0$ ordering were analyzed using X-ray diffraction (XRD) with Cu-K_α radiation (λ = 1.54056 Å). Microstructure was characterized by using transmission electron microscope (TEM, FEI Technai F20 and F30). The room temperature magnetic properties were measured by superconducting quantum interference device vibrating sample magnetometer (SQUID-VSM) with an applied magnetic field up to ± 70 kOe.

The present investigation reveals that the average FePt grain size as shown in Fig.1 decreases with increasing C volume fraction. The cross sectional TEM analysis confirmed the column growth of FePt grains without the formation of second layer of FePt grain. A minimum grain size of around 6.5 nm and the pitch distance of 7.6 nm is achieved with perpendicular coercivity of 4.4 Tesla (see inset of Fig.1). A systematic investigation on the effect of C volume fraction and graded structure on the degree of ordering, microstructure refinement with columnar growth and the resulting magnetic properties will be discussed in detail.

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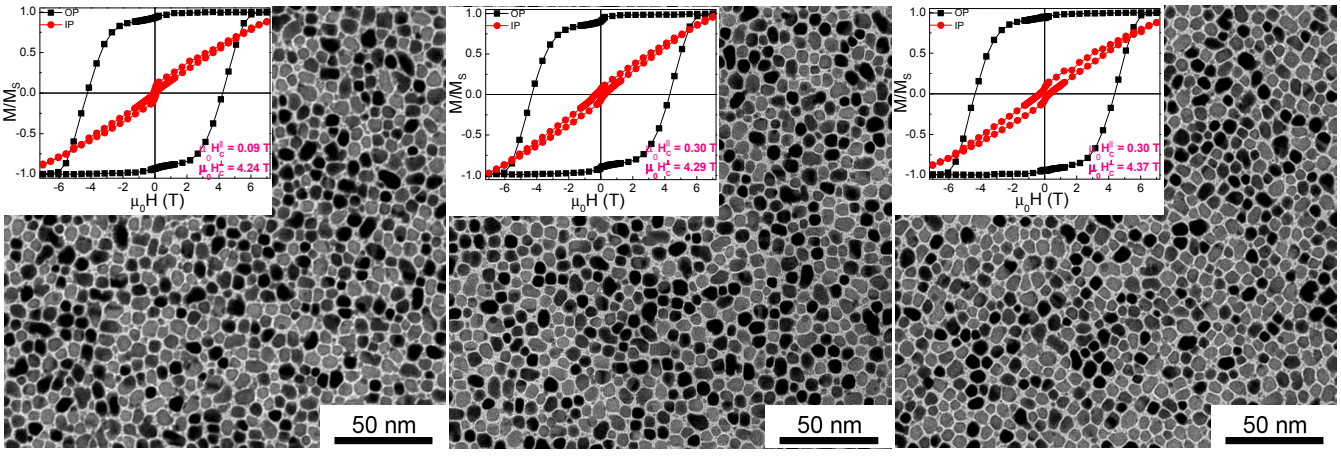

Fig.1: Plane view TEM images of FePt-C thin films with different carbon volume fraction in graded structure. Room temperature M-H loops are plotted in the inset.

$MgAl_2O_4$ 及び MgO 基板上に作製した FePt 薄膜の構造と磁気特性

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Structure and magnetic properties for FePt thin films prepared on $MgAl_2O_4$ and MgO substrates

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はじめに

磁気記録媒体は垂直磁気記録方式が用いられ飛躍的に記録密度が増加した。今後更なる高密度化が求められているが、高密度化により磁性粒子の縮小に伴う熱揺らぎの問題が深刻化する。次世代の磁気記録媒体の材料には高い結晶磁気異方性を有する材料が求められている。そこで注目を集めているのが $L1_0$ 型 FePt 規則合金であり、$L1_0$ 型 FePt 規則合金は高い結晶磁気異方性($K_u = 7.0\times10^7$ erg/cc)を有する事から次世代の磁気記録媒体材料として期待され多くの研究がなされている(1 ~ 3)。しかしながら、FePt 薄膜の磁化過程については未だ十分に解明されていない。そこで本研究では $L1_0$ 型 FePt 規則合金の格子定数の近い $MgAl_2O_4$ (MAO)(100)及び MgO(100)単結晶基板を用い、配向性及び規則度を調べ優れた FePt 薄膜を作製するため、その構造、表面形態および磁気特性を評価し、磁化過程について詳細に調べた。

実験方法

全ての試料は超高真空多元スパッタ装置を用いて作製した。到達真空度 8.5×10^{-7} Pa 以下及び Ar ガス圧 0.2 Pa にて成膜を行った。基板に MAO 単結晶基板及び MgO 単結晶基板上に基板温度 T_s = 700ºC にて FePt 層 (10 nm)成膜した。FePt 層の成を Fe_xPt_{100-x} (at. %) (x = 46.1 ~ 50.8)と変化させた。評価には膜組成は電子線マイクロアナライザ(EPMA)、結晶構造は X 線回折装置(XRD)、表面形状は原子間力顕微鏡(AFM)及び磁気特性は超伝導量子干渉計(SQUID)を用いて行った。

実験結果

全ての試料において X 線回折パターンより $L1_0$ 型 FePt 相の基本反射ピークである FePt(002)及び超格子反射ピークである FePt(001)、FePt(003)が観察された。Fig. 1 に Fe の組成を変化させた時の a 軸のグラフを示す。a 軸の格子面間隔はいずれの単結晶基板においても $Fe_{47.2}Pt_{52.8}$(at. %)の際に面間隔が最大を示し、それ以降 Fe の組成が増加するに伴い面間隔が縮小することが得られた。Fig. 2 に Fe の組成を変化させた時の保磁力のグラフを示す。保磁力は MAO 単結晶基板においては Fe の組成が増加するに伴い増加し、$Fe_{48.3}Pt_{51.7}$ (at. %)の時に最大 54.2 kOe が得られた。MgO 単結晶基板においても同様に Fe の組成が増加するに伴い増加し、$Fe_{49.3}Pt_{50.7}$ (at. %)の時に最大 57.8 kOe が得られた。また、いずれの基板においてもさらに Fe の組成が増加するに伴い保磁力の減少が確認された。

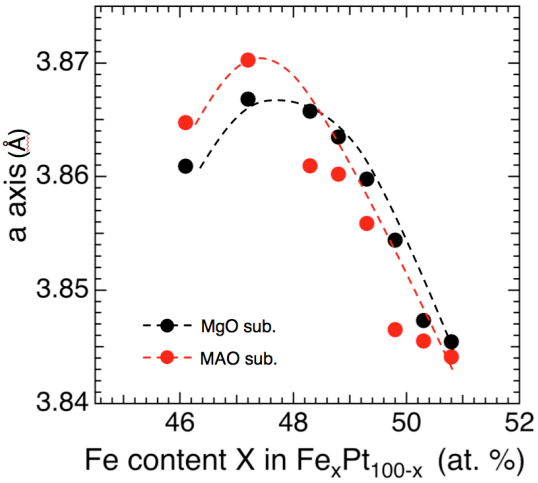

Fig. 1. a axis as a function of Fe content.

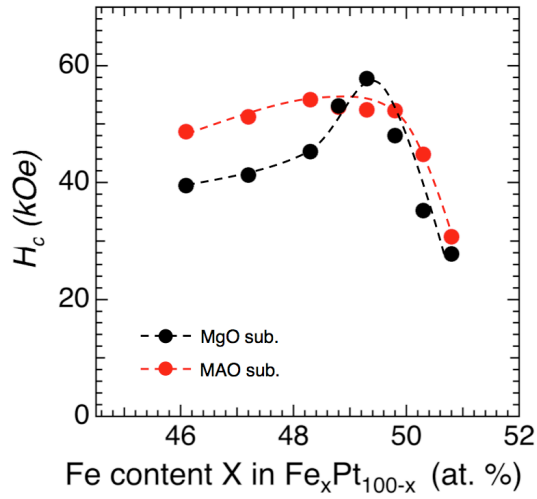

Fig. 2. H_c as a function of Fe content.

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