Preparation of Bi-iron-garnet Films on Platinized Si Substrate with Bi-substituted Y-iron-garnet Template Layer

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Bi-iron garnet (BIG) films were prepared using Bi-substituted Y-iron-garnet (Bi:YIG) template layer on platinized Si. First, Bi:YIG template layer was prepared on the platinized (100)Si (Pt/Ti/SiO_2/Si) by PLA deposition and successive rapid thermal annealing (RTA). Then $Bi_3Fe_5O_{12}$ film was grown on the template layer by PLA. The $Bi_3Fe_5O_{12}$ film deposited was found to have a garnet (BIG) phase by X-ray diffraction measurement. For the BIG film, the saturated magnetization and the Faraday rotation were characterized and discussed.

Keywords: Bi-iron garnet (BIG), Platinized Si substrate, Pulsed laser ablation, Magnetization, Faraday rotation

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1. Introduction

Amorphous rare-earth transition metals, which are conventionally employed for the magneto-optical (MO) recording, have some problems; a chemical instability due to rapid oxidation, a small MO effect, etc. On the other hand, Bi-substituted rare-earth iron-garnet ($Bi_xRe_{3-x}Fe_5O_{12}$; Bi:RIG) films have attracted much attention for the MO recording media with a high memory density.[1] This is mainly because the MO effect of this material is exceptionally large in the short wavelength region, and the chemical stability is excellent in the atmospheric environment. They have, however, a problem of the low read-out signal-to-noise ratio due to grain boundary scattering in the polycrystalline films.[2]

For reducing the light scattering, it is attractive to reduce crystalline size below the wavelength employed for reading.[2] In addition, for enhancing the MO effect, employing the completely Bi-substituted iron garnet with the composition of $Bi_3Fe_5O_{12}$ (BIG) is an attractive alternative[3,4] Here, the formulae of $Bi_xRe_{1-x}Fe_5O_{12}$ and $Bi_3Fe_5O_{12}$ do not necessarily mean the garnet structure but just the cation composition. Thus, for instance, the BIG means the garnet structure with the composition of $Bi_3Fe_5O_{12}$. Although the BIG is known to be thermodynamically unstable, the BIG film was reported to be grown on the single crystalline garnet substrate by ion beam sputtering.[4] So far, there is no report on the preparation of BIG films on non-garnet substrates.

Pulsed laser ablation (PLA), or pulsed laser deposition (PLD), has been attracting much attention on preparation of multicomponent oxide films with superior properties. Especially, we have prepared successfully lead zirconate titanate (PZT) thin films and Bi:RIG thin films by using PLA.[5,6] The most distinguished feature of the PLA is the film deposition with the composition same as the target[5,6].

In this article, preparation of BIG films on non-garnet substrates using template layers by PLA will be described. As the non-garnet substrate, platinized (100)Si ($Pt/Ti/SiO_2/Si$) substrates were employed using Bi-substituted Y-iron-garnet (Bi:YIG) template layers prepared by rapid thermal annealing (RTA).

Platinized (100)Si ($Pt/Ti/SiO_2/Si$) substrate was employed for the following reasons. (1) One third of the lattice constant of Bi:YIG (～ 1.2 nm) is close to that of Pt (a=0.3923 nm). (2) The thermal expansion coefficient of YIG is 8.13×10^{-6} /deg and that of Si is 2.6×10^{-6} /deg. The large difference in the thermal expansion coefficient between Bi:YIG and Si can be used for films with perpendicular magnetic

anisotropy using stress-induced magnetic anisotropy.[7] (3) The high reflectivity of Pt for visible light is preferable for read-out memory through the MO effect in the Kerr configuration. (4) It is easy to prepare the substrate with large area. The substrate is often used for the preparation of PZT films because of the oxidation-resistant bottom electrode for the capacitors.

It is, however, expected to be difficult to prepare the BIG film directly on the Pt layer, as described in detail below. A template layer for the preparation of BIG phase is required on Pt/Ti/SiO_2/Si. Recently, we obtained the garnet phase of $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ on Corning 7059 glass with successive RTA.[7] This material can be used as the template layer because it is thermodynamically stable.

2. Experimental

A schematic view of our equipment for the preparation of films by PLA was described in our previous paper.[8] The film deposition was carried out by PLA of sintered targets in the vacuum chamber filled with an O_2 gas of 27 Pa. We used an ArF excimer laser (SHIBUYA SQL2240, wavelength 193 nm, pulse width 10 ns, repetition frequency 5 Hz). The laser beam was focused by a quartz lens and directed to the target with the incident angle of 45 deg. In order to ablate fresh target surface the laser beam was continuously scanned on the target surface. We prepared targets by sintering the pellets of $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ and $Bi_3Fe_5O_{12}$. The substrate was (111)Pt/(001)Ti/SiO_2/(100)Si. Pt and Ti were coated by sputtering on the thermally oxidized (100)Si. The Ti layer was used because of a good adhesion of Pt to the oxidized Si. The thicknesses of Pt, Ti and SiO_2 layers were about 300 nm, 100 nm and 100 nm, respectively. For comparison, (111)Gd-Ga-garnet (GGG) and MgO substrates were also employed.

For the preparation of Bi:YIG template layer, the laser fluence was varied from 3.3 to 5 J/cm^2 and the substrate temperature T_s was 520 ℃. There is no essential difference in the film structure and quality originating from the difference in the laser fluence. RTA of the deposited Bi:YIG film was performed in an atmospheric ambient at 750 ℃ for 5 or 10 min. For the preparation of BIG films, the laser fluence was 3.3 J/cm^2 and T_s was varied from 540 to 600 ℃.

X-ray diffraction (XRD) measurement was carried out with Cu K_α radiation. The magnetic property was characterized by a vibrating sample magnetometer (VSM) at room temperature. The Faraday rotation angle vs. applied

magnetic field H was measured using the reflected light from the Pt surface, i.e., in the Kerr configuration. The measurement was performed at room temperature and with the wavelength of λ=633 nm. The X-ray photoelectron spectroscopy (XPS) was carried out for measuring the depth profile of the film composition. For analysis of the entire film composition, the electron probe microanalysis (EPMA) was also employed.

3. Results and discussion

3.1 Preparation of $Bi_3Fe_5O_{12}$ films on GGG or MgO substrates

First of all, the $Bi_3Fe_5O_{12}$ film was deposited on the GGG substrate at T_s=540 ℃ and with a laser fluence of 3.3 J/cm^2 by PLA. Figure 1 shows the XRD spectrum of the $Bi_3Fe_5O_{12}$ film on the (111)GGG substrate. Only (444) diffraction peak from BIG phase with a high intensity was observed, suggesting the BIG film [111] highly-oriented to the substrate normal is grown on the (111)GGG substrate. It is quite easy to obtain BIG phase on GGG by PLA, similarly to the ion beam sputtering.[4)] Additional experiments show that the deposition by PLA at a lower temperature of 475 ℃ gives rise to diffractions showing a phase other than the garnet phase even on the GGG substrate. The depositions at 540 ℃ over the range of the O_2 pressure of 13 - 130 Pa bring about the formation of the BIG single phase with a [111] orientation along the [111] of GGG.

As a non-garnet substrate, (100)MgO was employed for the preparation of BIG films by PLA. First, $Bi_3Fe_5O_{12}$ films were deposited directly on (100)MgO substrates which are often used for the preparation of high-T_c $YBa_2Cu_3O_x$ (YBCO) films. But no crystallized phase was observed in the as-deposited film by XRD measurements. Then RTA was performed at 750 ℃ for crystallization. It was still impossible to obtain the garnet phase because of the reevaporation of Bi. The Bi-deficiency in the RTA treated film was confirmed by EPMA. Preparation of $Bi_3Fe_5O_{12}$ film was performed directly on the Pt/Ti/SiO_2/Si at T_s ranging from 540 ℃ to 600 ℃. However, it was also impossible to obtain the garnet phase. XPS measurements for this sample revealed that there is no large deficiency of Bi atoms and no large interdiffusion between $Bi_3Fe_5O_{12}$ and Pt/Ti/SiO_2/Si, suggesting that the film composition does not largely deviate from the composition of $Bi_3Fe_5O_{12}$ on the Pt/Ti/SiO_2/Si.

For the preparation of the garnet phase, YBCO was employed as a template

layer because the YBCO film was reported to be grown on a garnet (Y-Al garnet : YAG) substrate with the lattice parameter of 1.2009 nm.[9] A highly [001]-oriented YBCO film was grown on the (100)MgO substrate by PLA, and then the $Bi_3Fe_5O_{12}$ film was deposited on it. However, the film shows diffractions from orthoferrite of $BiFeO_3$. The magnetization measurement revealed that the magnetization is far below that of the BIG.

These results lead us to a conclusion that it is very difficult to prepare BIG on non-garnet materials, such as MgO and YBCO, because of the instability of BIG on the non-garnet structure.

3.2 Preparation of Bi:YIG template layer on platinized (100)Si

Figure 2 shows the XRD spectra for an as-deposited $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ film prepared at a laser fluence of 5 J/cm^2 and at T_s=520 ℃ (a), a $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ film treated by RTA at 750 ℃ for 5 min (b) and a $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ film treated by RTA at 750 ℃ for another 10 min (c). The substrates are Pt/Ti/SiO_2/Si. Closed triangles represent the diffractions from garnet phase. Diffractions from Pt/Ti/SiO_2/Si are labeled by the element name. From these figures it is found that the RTA gives rise to a formation of the garnet phase while the as-deposited film has almost no garnet phase. The similar result of RTA was observed for $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ films on the Corning 7059 glass.[7] Hereafter, the $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ film crystallized with garnet phase will be designated the Bi:YIG film or template layer.

The magnetization measurement was carried out for the Bi:YIG film. Figure 3 shows the magnetization hysteresis loop obtained by using the VSM for the Bi:YIG film treated by RTA at 750 ℃ and for 5 min on Pt/Ti/SiO_2/Si. Magnetic field H is applied to the film either in parallel with the film plane (a) and perpendicularly to the film plane (b). The saturation magnetization was found to be about 1400-1500 G, which was close to the value of well-crystallized Bi:YIG.[10] However, the film reaches the saturated state more easily by increasing the parallel applied field than the perpendicular filed, suggesting that the present Bi:YIG film has a magnetic easy-axis parallel to the film plane.

Figure 4 shows the XPS depth profile for the Bi:YIG film treated by the RTA at 750 ℃ for 15 min. The concentration was derived from Bi 4d, Y 3p, Fe 2p, O 1s and Pt 4f signals. It should be noted that preferential sputtering probably causes

the off-stoichiometry in the Bi:YIG template layer. Although a small diffusion of Fe atoms into the Pt layer was observed in the figure, almost no diffusion of Bi, Y and O atoms into the Pt layer take place. At present, the influence of the diffusion of Fe on the formation of Bi:YIG has not been clarified yet. Anyway there is no large interdiffusion as can be seen from the figure. For enhancing the stress-induced magnetic anisotropy perpendicularly to the film plane, $Bi_2Dy_1Fe_5O_{12}$ (Bi:DIG) film was prepared on Pt/Ti/SiO_2/Si by PLA and the successive RTA similarly to the Bi:YIG film preparation was carried out.[7] This film, however, has also the magnetic easy-axis parallel to the film plane. This is probably caused by the formation of cracks in the film plane induced by the excessively large difference in the thermal expansion coefficient.

3.3 Preparation of BIG film on platinized (100)Si using Bi:YIG template layer

First, the Bi:YIG template layer was prepared by PLA and successive RTA (T_s=750 ℃ and 5 min), as described in the previous subsection. For the deposition of template layer, a laser fluence of 3.3 J/cm^2 was employed. On this template layer, $Bi_3Fe_5O_{12}$ films were deposited by PLA at substrate temperatures ranging from 540 to 600 ℃ and at an O_2 pressure of 27 Pa. Figure 5 shows the XRD spectra for a Bi:YIG template layer treated by the successive RTA (a), a $Bi_3Fe_5O_{12}$ film deposited at 540 ℃ on the Bi:YIG template layer (b) and a $Bi_3Fe_5O_{12}$ film deposited at 600 ℃ on the Bi:YIG template layer (c). Open circles in Fig. 5 (a) represent diffractions from the Bi:YIG template layer. As can been seen from Figs. 5(b) and 5(c), after the deposition of $Bi_3Fe_5O_{12}$ films a new diffraction appears near the (420) diffraction of Bi:YIG around 32 deg in the low angle side. The incorporation of Bi^{3+} causes a strong lattice expansion because of its large ionic radius (0.1132 nm).[4] The expansion rate for $Bi_xY_{3-x}Fe_5O_{12}$ with Bi-incorporation is 0.00828 nm /(1 Bi atom/formula unit), which will give a lattice parameter of 1.2624 nm for BIG.[4] A similar result was reported by Okada et al.[11] These mean that the (420) diffraction of BIG appears at lower angle than that for Bi:YIG if BIG phase is formed. Thus, we conclude that the diffraction represented by the closed triangle at lower angle than the open circle is the (420) diffraction from BIG phase. Other small diffractions are also from the garnet phase while it is difficult to distinguish the BIG from the Bi:YIG because of the poor signal to noise ratio.

For further confirmation of the formation of BIG phase, the magnetization

measurement was performed for these BIG/Bi:YIG/Pt/Ti/SiO_2/Si samples by VSM. Before calculating the magnetization using the total film thickness of BIG and Bi:YIG, the values of magnetic moment for Bi:YIG and BIG/Bi:YIG samples were compared. The result showed that the magnetic moment was obviously increased by the deposition of BIG film. This is consistent with the XRD result that the ferrimagnetic BIG film was grown on the Bi:YIG template layer. Figure 6 shows the magnetization hysteresis loops for BIG/Bi:YIG samples in the applied field H perpendicular to the film plane. The BIG films were prepared at 540 ℃ (a) and 600 ℃ (b). The magnetizations were averaged ones calculated using the magnetic moment and the total film thickness of BIG and Bi:YIG. Comparison of Figs. 6(a) and 6(b) suggest that it is easier to saturate the magnetization for the BIG(T_s=600 ℃)/Bi:YIG sample than for BIG(T_s=540 ℃)/Bi:YIG sample. Comparison of the magnetization hysteresis loop in the applied magnetic field H parallel and perpendicular to the film plane tells us that both the samples have magnetization easy axis in the film plane rather than the film normal.

The Faraday rotation angle was measured for these samples. Figure 7 shows the Faraday rotation angle at λ=633 nm vs. applied magnetic field H perpendicular to the film plane for Bi:YIG template layer (a), BIG(T_s=540℃)/Bi:YIG (b) and BIG(T_s=600℃)/Bi:YIG. It should be noted that the sensitivity for the spectra is different from each other. The saturated Faraday rotation angle $4\theta_f$ is shown by the bar in the right hand side. It is clearly seen from these figures that the deposition of BIG film gives rise to a remarkable increase in $4\theta_f$. The Faraday rotation F was calculated for each layer separately. Table Ⅰ summarizes that the Faraday rotation F derived from Fig. 7 for the Bi:YIG film in the Bi:YIG/Pt/Ti/SiO_2/Si sample, the BIG film in the BIG(T_s=540℃)/Bi:YIG/Pt/Ti/SiO_2/Si sample and the BIG film in the BIG(T_s=600℃) /Bi:YIG /Pt/Ti/SiO_2/Si sample. As shown in Table Ⅰ, F for both the BIG films is of the order of 10^4 deg/cm, suggesting that the deposited film obviously has the garnet phase. The value of F is, however, small compared with that for BIG prepared on a single crystal garnet substrate. For instance, Okuda et al. reported that the BIG film prepared on a $(GdCa)_3(GaMgZr)_5O_{12}$ substrate shows a remarkably large Faraday rotation F of -7.2×10^4 deg/cm.[3] at λ=633 nm. Besides, in the present measurement, some Faraday loops with irregular shapes were also obtained probably owing to inhomogeneity of these samples. These results imply that the quality of the present BIG films are inferior to that for the epitaxial

film, suggesting that a further optimization is required.

Thus, the XRD and the Faraday rotation angle measurements revealed that the garnet structure was obtained by PLA of the $Bi_3Fe_5O_{12}$ sintered target using Bi:YIG template layer on the Pt/Ti/SiO_2/Si substrate. It is, however, necessary to check the possibility that Y atoms in Bi:YIG template layer diffuse into $Bi_3Fe_5O_{12}$ layer and thermodynamically stable $Bi_xY_{3-x}Fe_5O_{12}$ films were formed with the Y atoms with the garnet phase. To clarify this point the XPS depth profile was measured for the BIG/Bi:YIG sample. Figure 8 shows the XPS depth profile for Bi 4d, Y 3d, Fe 2p and O 1s components for the BIG(T_s=540℃)/Bi:YIG/Pt/Ti/SiO_2/Si sample. It should be noted that preferential sputtering probably causes the off-stoichiometry in the BIG and Bi:YIG regions. Y atoms were detected only in the bottom Bi:YIG template layer and they were not detected through the whole BIG region. From these data we can see that almost no interdiffusion of Y atoms takes place in the BIG region, supporting the result that the garnet structure was obtained by PLA of the $Bi_3Fe_5O_{12}$ target using Bi:YIG template layer on Pt/Ti/SiO_2/Si substrate.

4. Conclusion

Following results were obtained: (1) It is very difficult to prepare BIG on non-garnet materials, such as MgO and YBCO, because of the instability of BIG on the non-garnet structure. (2) $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ films were deposited on the Pt/Ti/SiO_2/Si by PLA and were treated by RTA, resulting in the garnet structure of $Bi_{1.5}Y_{1.5}Fe_5O_{12}$. (3) $Bi_3Fe_5O_{12}$ films with the garnet phase (BIG) were grown on non-garnet Si substrates using the above Bi:YIG template layers. (4)The crystal structure and the MO quality for the present BIG films were inferior to those for the epitaxial BIG films on single crystal substrate.

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TABLE CAPTION

Table Ⅰ Faraday rotation of samples.

FIGURE CAPTIONS

Fig. 1 XRD spectrum of the BIG film on (111)GGG substrate.

Fig. 2 XRD spectra of as-deposited $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ film at T_s=520 ℃ (a), $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ film treated by RTA at 750 ℃, (b) for 5 min and $Bi_{1.5}Y_{1.5}Fe_5O_{12}$ film treated by RTA at 750 ℃ for another 10 min (c) on Pt/Ti/SiO_2/Si. Diffractions from Pt/Ti/SiO_2/Si are indicated by each element name. Closed triangles show diffractions from garnet phase of $Bi_{1.5}Y_{1.5}Fe_5O_{12}$.

Fig.3 Magnetization hysteresis loop for a Bi:YIG film treated by RTA at 750 ℃ and for 5 min on Pt/Ti/SiO_2/Si. Magnetic field H is applied to the film in parallel with the film plane (a) and perpendicularly to the film plane (b).

Fig.4 XPS depth profile of the concentration for Bi:YIG film treated by RTA at 750 ℃ for 15 min on Pt/Ti/SiO_2/Si. Concentrations of each element are indicated by Bi 4d, Y 3p, Fe 2p, O 1s and Pt 4f.

Fig.5 XRD spectra for Bi:YIG film treated by RTA (a), $Bi_3Fe_5O_{12}$ film deposited at 540 ℃ on Bi:YIG (b) and $Bi_3Fe_5O_{12}$ film deposited at 600 ℃ on Bi:YIG (c). Open circles represent diffractions from Bi:YIG template layer. Closed triangle shows the diffractions from garnet phase of $Bi_3Fe_5O_{12}$.

Fig. 6 Magnetization hysteresis loops for BIG/Bi:YIG samples in the applied field H

perpendicular to the film plane. BIG films were prepared at 540 ℃ (a) and 600 ℃ (b). The magnetizations were averaged ones calculated using the total film thickness of BIG and Bi:YIG.

Fig. 7 Faraday rotation angle vs. applied magnetic field for Bi:YIG (a), BIG (T_s=540℃)/Bi:YIG (b) and BIG(T_s=600℃)/Bi:YIG.

Fig. 8 XPS depth profile of the concentration for the BIG(T_s=540℃)/Bi:YIG/Pt/Ti/SiO_2/Si sample. Concentrations of each element are indicated by Bi 4d, Y 3d, Fe 2p and O 1s.

	Bi:YIG	BIG in BIG(T_s=540℃) /Bi:YIG	BIG in BIG(T_s=600℃) /Bi:YIG
Faraday rotation F (deg/cm)	-2.3×10^4	-1.3×10^4	-2.4×10^4

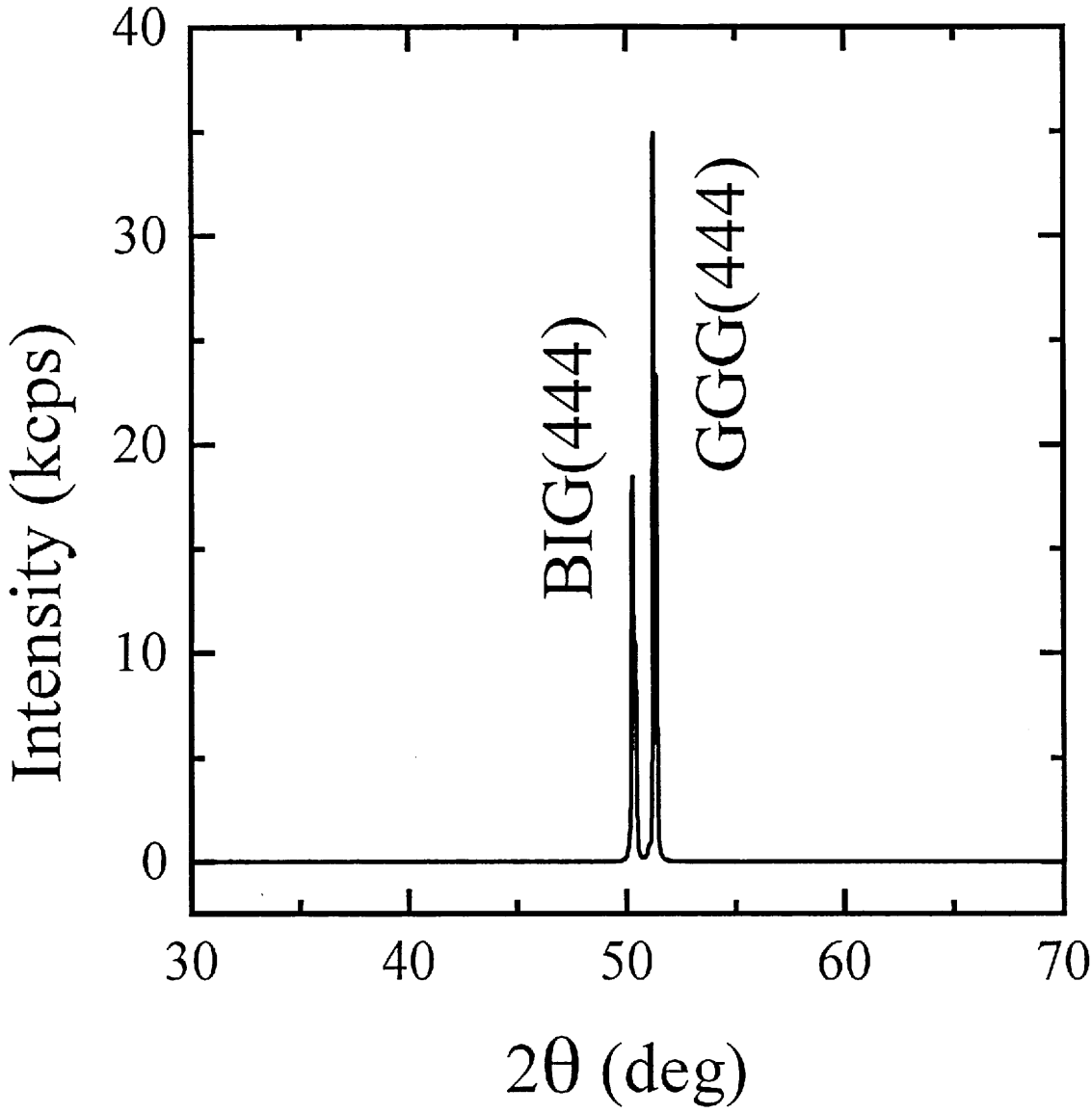


Fig.1 Yamazaki et al

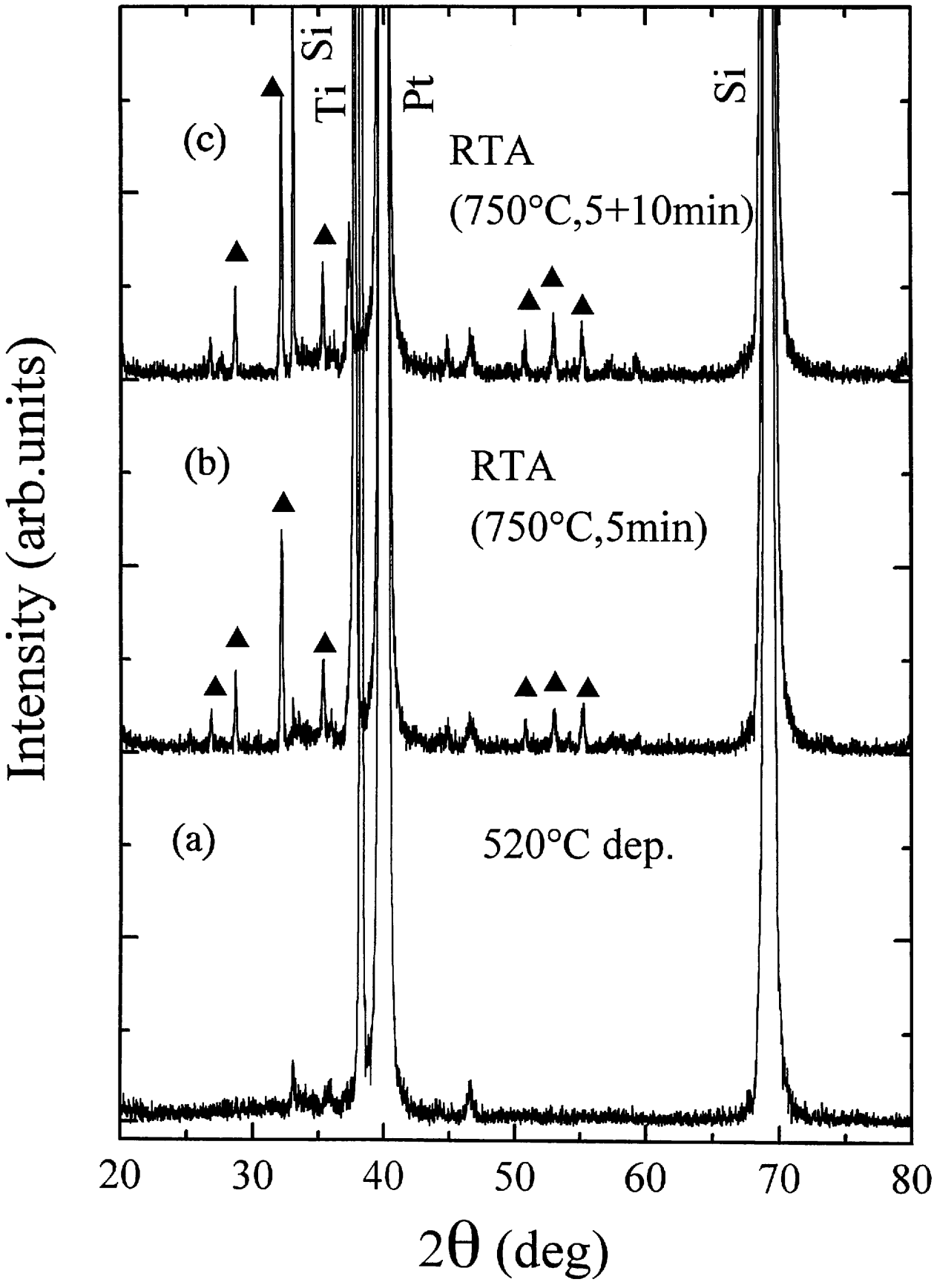
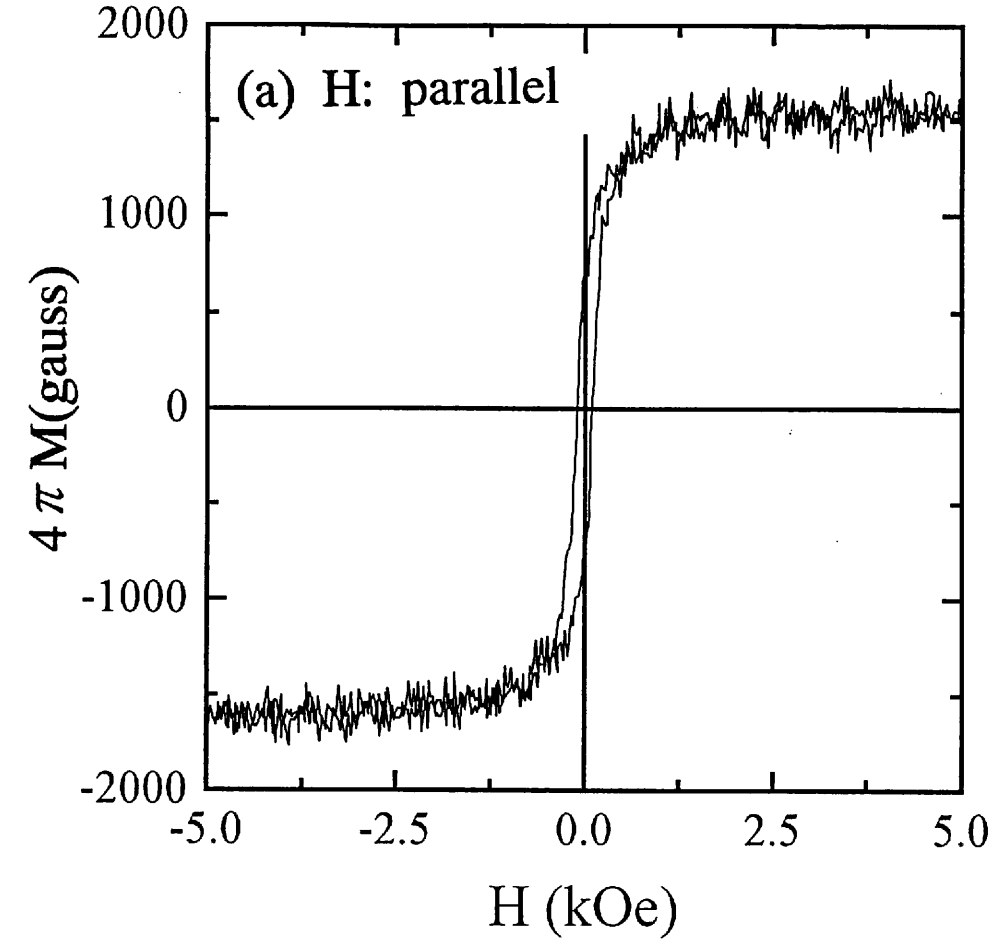
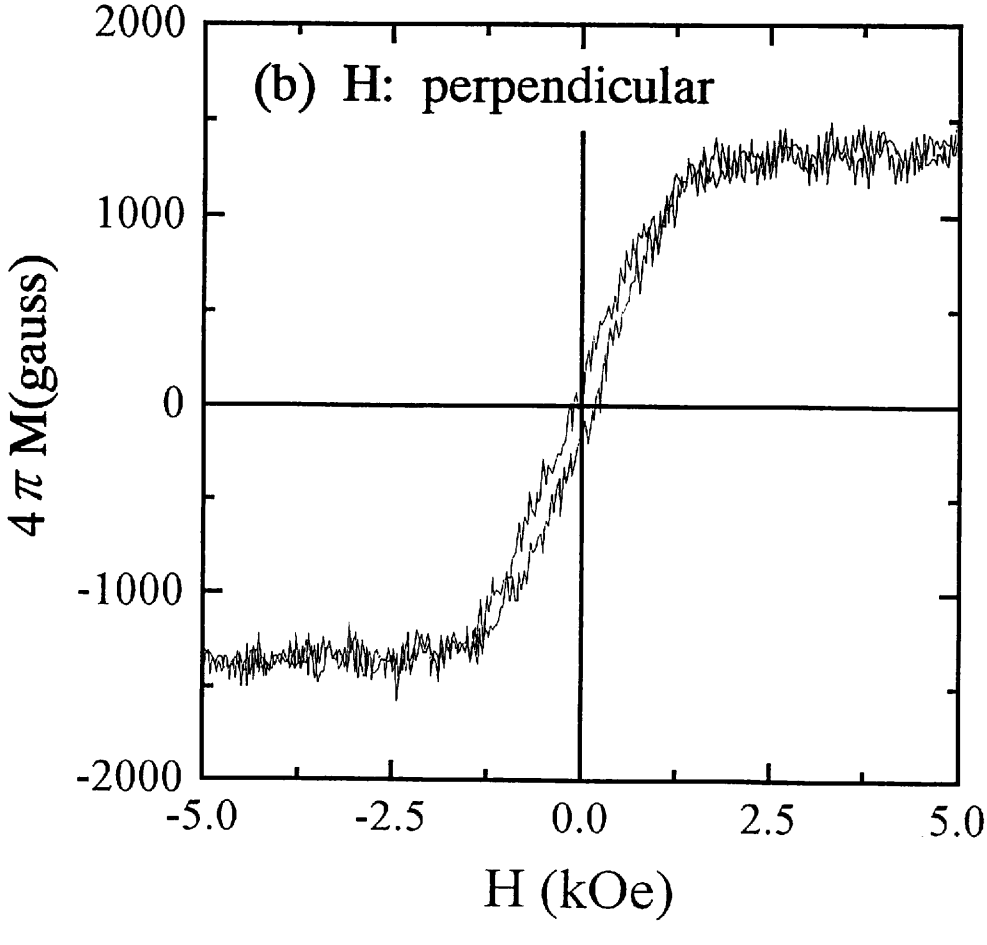


Fig. 2 Yonezawa et al


(a) H: parallel
2000
1000
0
-1000
-2000
-5.0
-2.5
0.0
2.5
5.0
4 π M(gauss)
H (kOe)


(b) H: perpendicular
2000
1000
0
-1000
-2000
-5.0
-2.5
0.0
2.5
5.0
4 π M(gauss)
H (kOe)

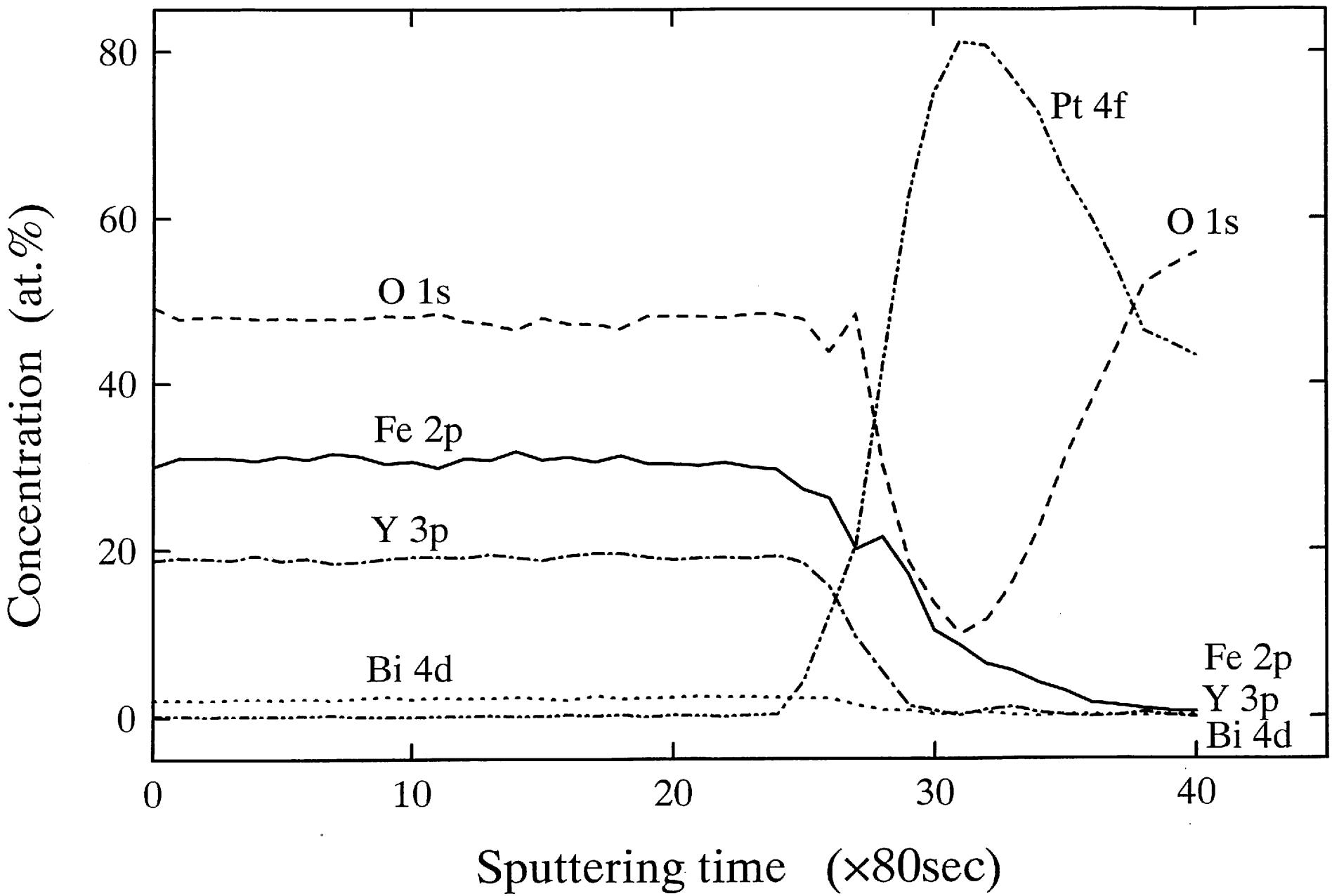


Fig 4 Yamazaki et al

(10cm)

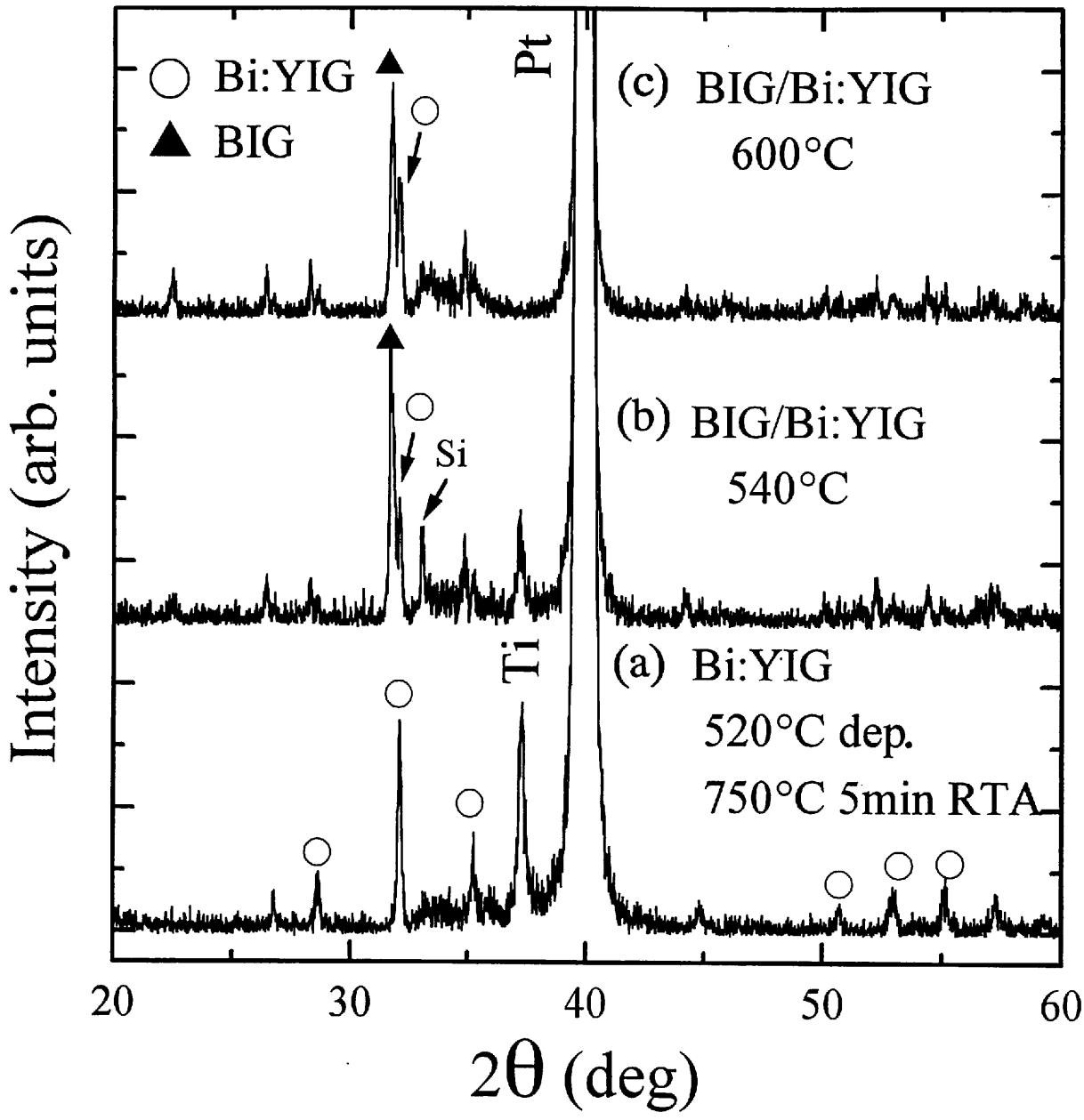
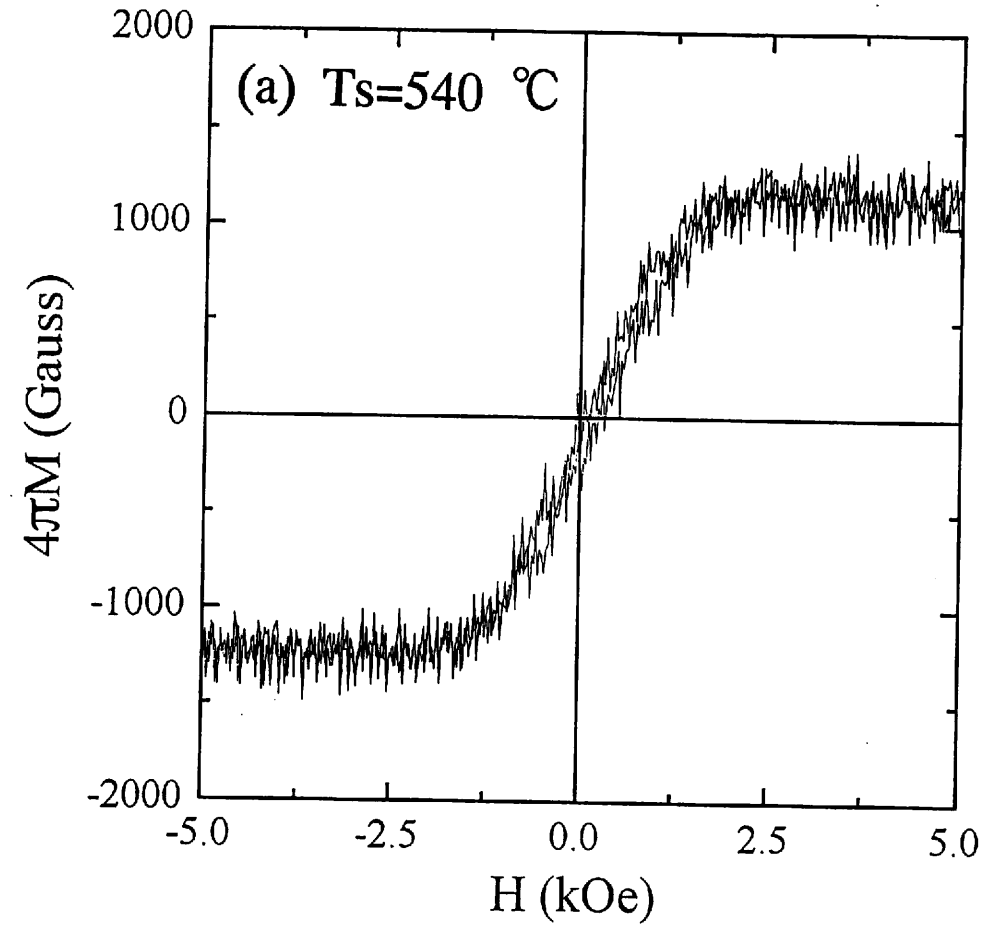


Fig. 5 Yonezawa et al.



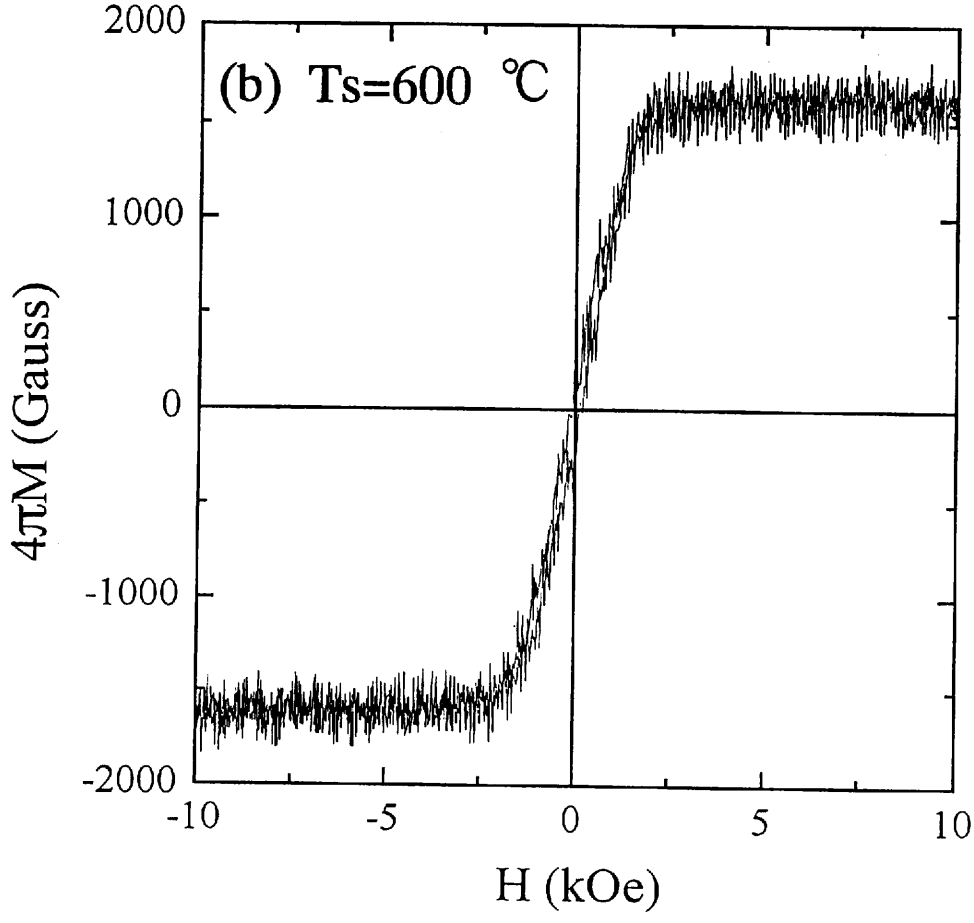


Fig 6 Yonezawa et al.

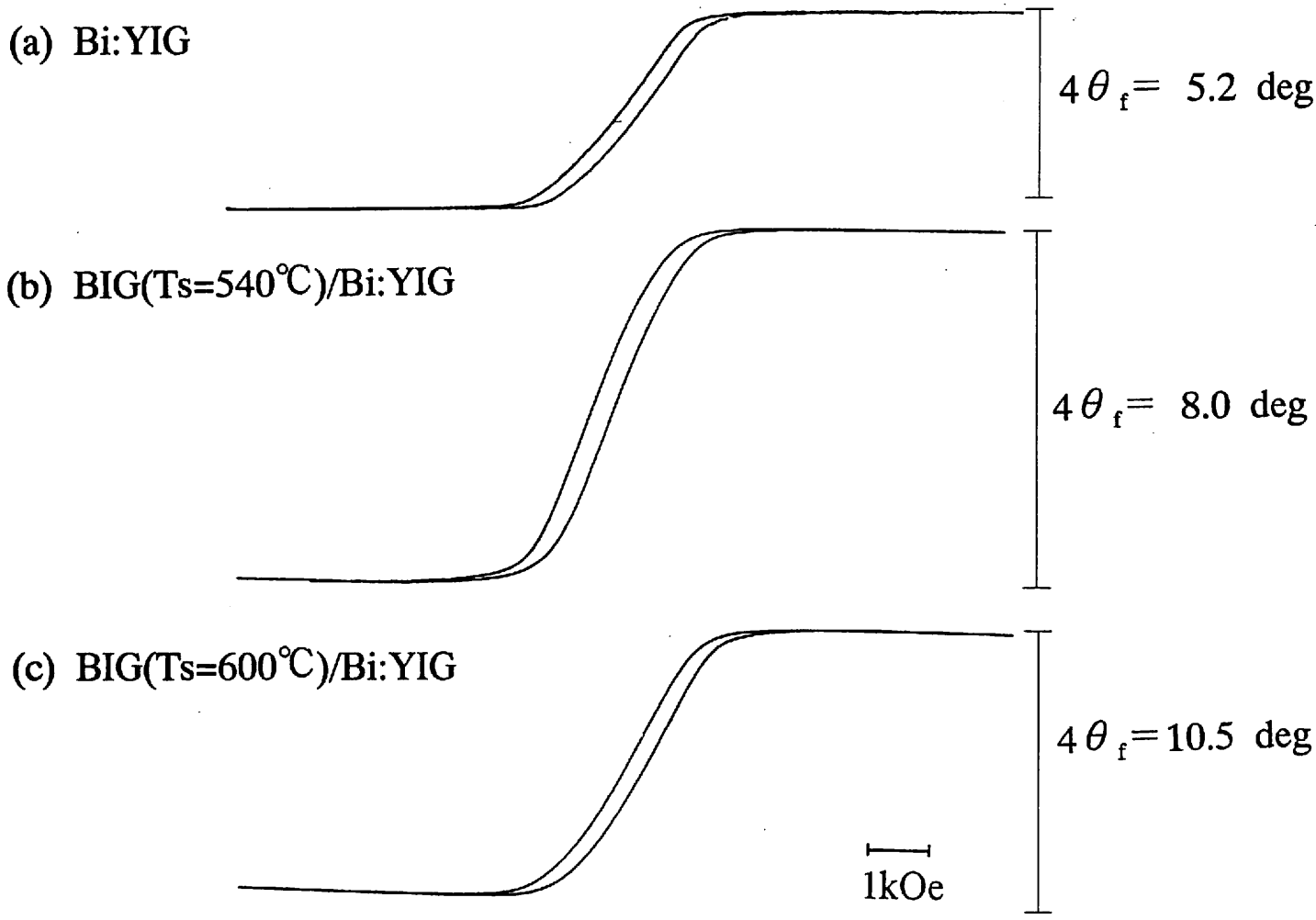
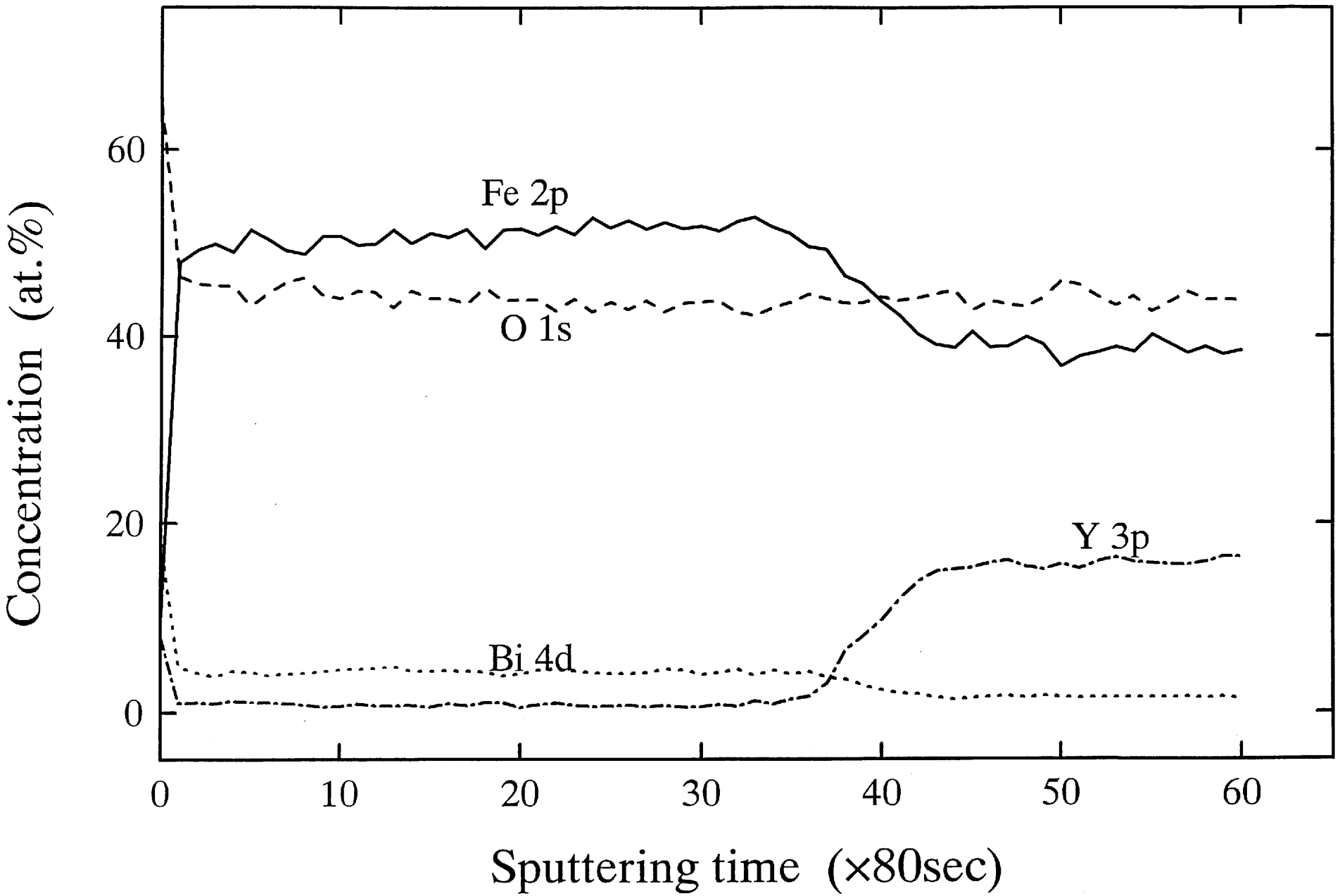


Fig. 7 Yonezawa et al


Concentration (at.%)
60
40
20
0
Fe 2p
O 1s
Y 3p
Bi 4d
0
10
20
30
40
50
60
Sputtering time (×80sec)

(10 cm)