RELATIVE SPUTTERING RATES FOR OXIDES VERSUS IRON USING 1 KEV ARGON IONS

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Abstract

Relative sputtering rates versus Fe for depositing oxide films of MgO, Al_2O_3, TiO_2, Cr_2O_3, FeO, Fe_2O_3, and CuO on silicon wafer using argon ions were measured by Auger electron spectroscopy. These iron and oxide films were prepared by depositing the oxide materials onto the silicon wafers using magnetron sputtering. These sputtering rates were compared with previous results on the sputtering rates for elements.

1. Introduction

The characterization of thin oxide films is a prominent subject in the surface analysis of solids. In the surface analysis of iron and steels, for instance, it is desirable to quantitatively characterize different oxide films on steels, which affect surface properties. These oxide films sometimes consist of several elements, which can be in-depth analyzed by a combination of Auger electron spectroscopy (AES) or X-ray photoelectron spectroscopy (XPS) and inert gas ion sputtering, being an indispensable technique for in-depth analysis[1-3]. However, there are several problems in these analysis: depth resolution, changes in sputtering rates for materials, and so on. To date, sputtering yields for pure elements have been investigated extensively and summarized[4,5]. However, sputtering behavior should be examined for different materials. The aim of this communication, therefore, is to report on the relative sputtering rates for oxide films versus that for an iron film. The specimens were produced on silicon wafers using a magnetron sputtering method. The in-depth profiles was surveyed using AES.

2. Experimental

Oxide films of about 100 nm thickness were deposited on silicon wafers using magnetron sputtering equipment. The oxides sputtered were MgO, Al_2O_3, TiO_2, Cr_2O_3, FeO, Fe_2O_3, and CuO. The specimens were prepared by Kyodo international Inc. Precise thickness of the films were measured using a surface roughness meter, Dektak. The density of these oxide films are assumed to be same as those of bulk for simplicity. The sputtering profile was measured using an Auger electron spectroscopy apparatus with an ion gun. Argon ions were accelerated with 1 keV by means of the ion gun, to sputter an iron film and oxide films on the silicon wafers. The angle between the ion beam and the normal of a specimen plane was 55 degree, and AES spectra were acquired using an accelerating voltage of 5 keV. The sputtering rate per unit time was calculated from the thickness and time to reach a half of the total change in the Si LMM peak height.

3. Results and Discussion

Figure 1 (a) and (b) shows sputter profiles of an Fe film and an Fe_2O_3 film, respectively, in which the concentration

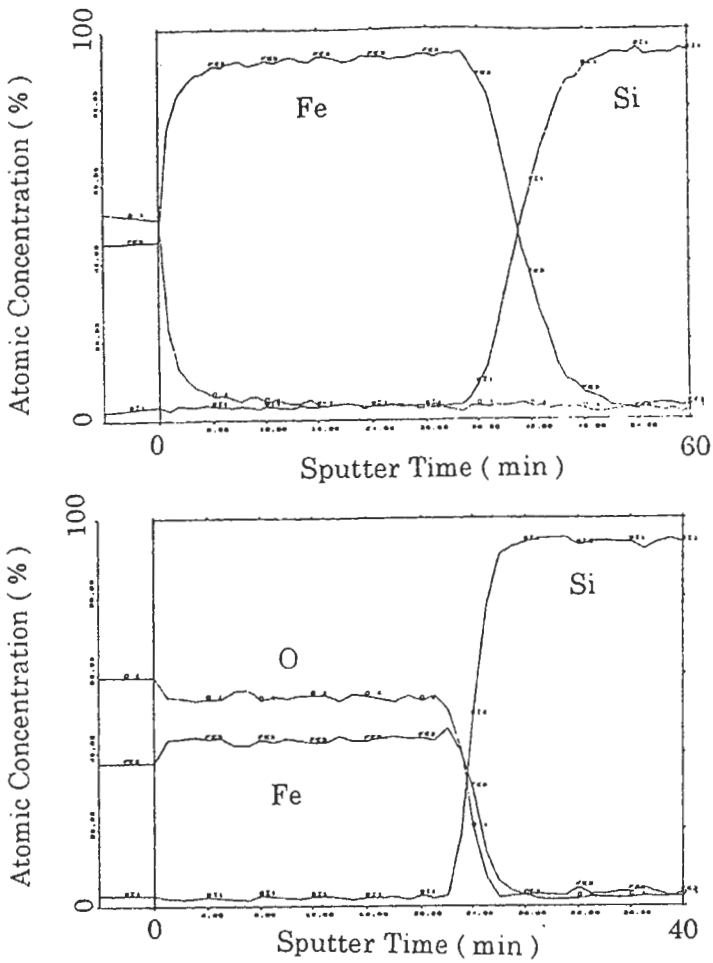

Figure 1 Sputter profiles of (a) an iron film and (b) an Fe_2O_3 oxide film.

corrected using relative sensitivity factors[6] is plotted as a function of sputter time. Since the shape of the Auger spectrum for an element may be affected by its chemical state, the correction used may not be enough. Therefore, it is difficult to compare the exact composition between the metal and the oxide. In spite of such limitation, the sputtering profiles provide information on the relative sputtering rate for the thin films by argon ions.

Based on data of sputter profiles of all oxide films, the relative sputtering rates for oxides versus iron were calculated, as summarized in Table 1, where the density of the film is same as that in bulk. The relative values include an error of about 10% from the reproductivity in measurements. The sputtering rate for oxide films was higher than that for iron. A ratio of sputtering rare of oxides to iron is between 1.34 and 2.66, while differences in rates between

Table 1 Relative sputtering rate (RSR) for examined oxides versus iron using 1 keV argon ions

	MgO	Al_2O_3	TiO_2	Cr_2O_3	FeO	Fe_2O_3	CuO
RSR	1.34	1.95	1.87	2.66	1.53	1.95	2.46

those oxides was within a factor of 2. Changes in sputtering behavior by materials may be caused by the density, chemical state, etc. of deposited elements.

Figure 2 shows the relative sputtering rates for elements and oxides as a function of the atomic number. The relative rates for elements were simply calcultated from values read from the figure plotted by Seah[4], whereas the data for oxides were obtained in the present work. The plots indicate that the sputtering rates for the oxide films were higher than that for iron, except for the case of magnesium. Although systematic changes are not obtained in this plot, it may be suggested that the sputtering rates strongly depend on the characteristic features of materials.

In summary, the present results show that the sputtering rates of oxide films were higher than those for iron by a factor 2.7, and that the sputtering rates of different oxides varied within a factor up to 2.

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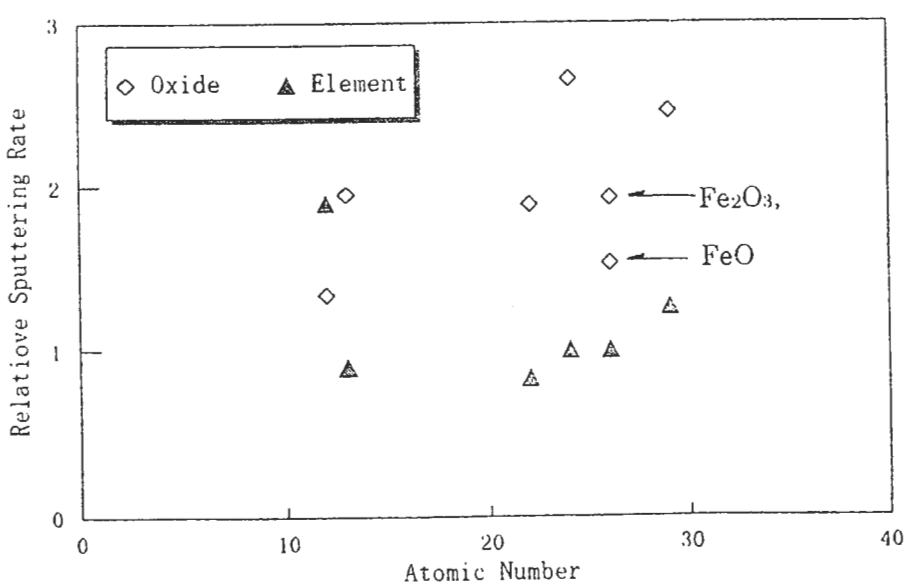

Figure 2 Relative sputtering rates for elements[4] and oxides as a function of the atomic number.

査読者との質疑応答

（査読者：関根／JEOL，本間／NTT）

本間：このような相対スパッタ速度のデータは，スパッタ・デプスプロファイリングの深さ軸の校正に有用なものです．今後，さらに，Arイオンの入射角やエネルギーに対する依存性が明らかにされれば，多くの人が利用できるデータになるのではないでしょうか．

ここでは，マグネトロンスパッタリングで形成された膜が用いられていますが，結晶形態はアモルファスと考えてよいのでしょうか．また，膜の密度はバルクの酸化物と同じなのでしょうか．

図２で文献４から読み出した金属のスパッタ速度との比較が行われています．文献４の値は垂直入射の場合のスパッタ収率で，Sigmundの式から計算したものです（実測値との差は少ないようです）．図２の金属の値は，単純にスパッタ収率比をプロットしたものでしょうか，それとも，密度を入れてスパッタ速度比に換算したものでしょうか．

酸化物のスパッタ速度の原子番号依存性が，Mgを除き，金属の場合に同じとされていますが，むしろ同じではないとみた方が妥当ではないでしょうか．酸化物が金属の場合と同じになる必然性は無いと思われます．

関根：1) Do you have any idea for the interpretation to that for magnesium pure metal shows a higher sputtering rate than its oxide in contrast with other elements examined?

2) You have listed up factors: density and chemical state as the the factors related to determining a sputtering rate of oxide films. As far as we see in Figure 2, at least the atomic number does not seem to relate it sharply. Do strength of chemical bonding and density of deposited film interprete the alternation of sputtering rate among oxides of different elements? For this, it will be comprehensive if sputtering rates are plotted agaist strength of chemical bonding or density.

著者：質問とうにたいして，全般的に回答したいと思います．

この実験の目的は，鉄を中心に金属と酸化物のスパッター速度を大まかに比較することです．というのも，いろいろな試料の分析をしていると大まかな金属と酸化物のスパッターに関する指針の必要性を感じているからです．ここで，大まかと言ったのは，標準となる試料のスパッター膜の実体がどこまではっきりしているのか自信がないからです．査読者の方からもご指摘がありましたように，膜の結晶性，密度等の実態が十分わかればよいのですが，その解析には結構な労力を必要としますし，それらの方法も十分確立されていないように思われます．私どもも他の方法によりその評価を試みておりますが，出てくる結果にはいろいろな面で任意性があります．したがって，本実験結果はスパッターを併用する表面分析における一つの目安となればよいと思っております．また，そのデータを基に二次的にデータを加工したり，他の性質と定量的に比較したりすることも可能でしょうが，そこでは任意性が増幅されますので，ここでは結果を示すにとどめました．

もっともなご質問に対し少々歯切れの悪いところもあるかもしれませんが，本実験結果を参考データとしてご覧いただければと存じます．