Review

Modification of Thin Film Properties by Sputtered Particles

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1. Introduction

The novelty of the functions provided by plasma-assisted physical vapor deposition films, together with their durability for practical use, is emphasized as the area where the thin film process has a significant impact. Remarkable advances have been made in recent years in science and technology of the thin film process for deposition. Up to the present, thin films have been studied from the viewpoint of the relationship between their structure and properties [1-3]. On the other hand, the commercial use of thin films has been growing at a surprisingly rapid rate for the last two decades in almost all the industrial fields such as electronics and optics. These films are practically formed by depositing materials onto a supporting substrate to build up a thin film thickness through a complicated thin film process rather than by thinning down bulk materials by simple methods such as mechanical polishing and ion milling.

Nowadays, thin film technology has been classified by the method of film formation such as physical vapor deposition (PVD) or chemical vapor deposition (CVD). In addition, the resulting films are also characterized by their chemical bondings such as organic or inorganic films. Therefore, it is essential to understand the elementary stages of formation in thin film process in which low starting materials are modified from bulk to thin films.

In this review paper, I first describe a new concept of the thin film process in material synthesis on the basis of ion-surface interaction during deposition. Some examples of practical applications of SiO_2 and Ni-Si-B films are given.

2. Thin Film Process

Irrespective of thin film preparation methods such as PVD and CVD, the thin film process comprises three elementary stages including decomposition, transport, and nucleation and growth mechanisms [4]. Fig. 1 shows the flow chart of the thin film process, where starting materials are successively modified to the resulting films. In the first stage, starting materials in the form of gas, liquid or solid are decomposed into various fragments of neutrals or ions in the form of atoms, molecules, clusters or powders by the external powers of plasma, laser, ion, microwave and thermal energies. The decomposed fragments thus formed will travel through the medium of gas or liquid and approach to the substrate. This phase is referred to as the transport stage in the thin film process. The Monte Carlo simulation

Keywords Thin film, Sputter deposition, Kinetic energy, Film properties

technique is helpful in understanding the mechanism of the transport stage. The chemical reaction between the transport medium and the decomposed fragments is also important in the reactive process of thin film formation. The decomposed fragments land on the substrate for nucleation and growth, which result in the formation of functional films. Thin film properties are strongly influenced at this final stage, because the energy of the decomposed fragments is dissipated in the very shallow surface region of the substrate. This dissipation of the energy of the decomposed fragments may enhance the surface migration of ad atoms, chemical reaction between landing fragments and adsorbed molecules, and finally reconstruction into the structures of the resulting films.

Consequently, we have possibilities of obtaining superior properties of materials in the form of thin films compared to bulk through the thin film process shown in Fig. 1.

On the other hand, the influence of the kinetic energy of particles in the thin film process on thin film formation ranges from simple substrate cleaning for enhanced adhesion to morphological changes and epitaxial growth. Peculiar features of sputter deposition technique are generally explained in terms of the energy of the sputtered particles which are decomposed and emitted as a result of high energy ion bombardment in plasma. A complex variety of processes simultaneously occurs whenever energetic particles interact with a substrate or growing films. For a comprehensive description of these processes, I refer to a number of excellent reviews in the literature [2, 3]. The sputtering process is discussed in view of the energy distribution of sputtered particles and some chemical effects on the substrate surface by high energy ion bombardment.

3. Ion-Surface Interaction Processes

The importance of ion-surface interaction processes in thin film deposition depends on

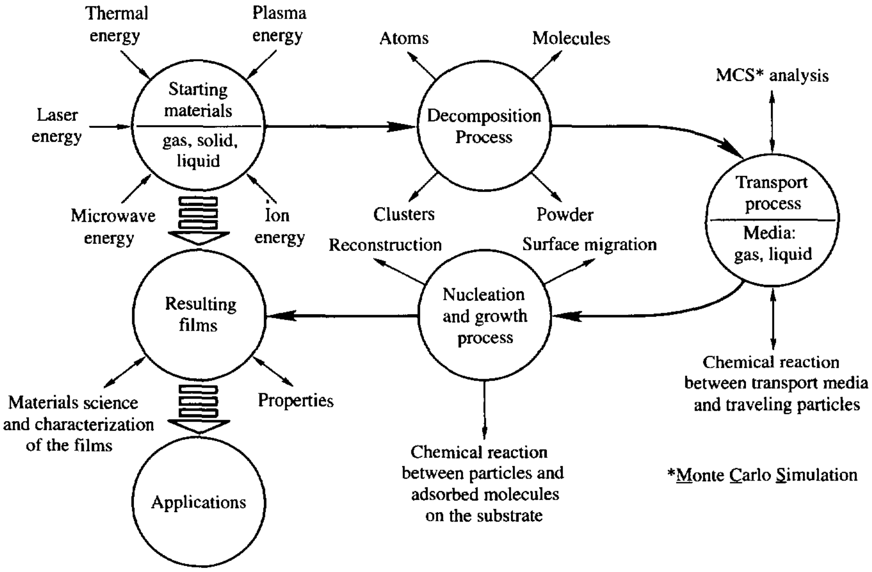


Fig. 1 Flow chart of the thin film process.

the particular deposition process employed. For a comprehensive description of these processes, a number of excellent reviews can be seen in the literature [5-7]. In the present discussion, the ion-surface interaction processes illustrated in **Fig. 2** are considered, where sputtering, ion reflection, penetration and trapping are briefly reviewed.

One of the principal effects during ion bombardment of a solid surface is the ejection or sputtering of the surface atoms. It is well understood that the energetic primary ions lose energy in a series of elastic and inelastic collisions with target atoms. The recoiling target atoms give rise to secondary collisions in a collision cascade. The energy of primary ions may eventually be transferred to the surface atoms which will be ejected if the surface binding energy is overcome. Sputtering results from collision sequences less than five atomic layers beneath the surface. These atoms, ejected as a result of sputtering, deposit onto a substrate which may be close to the target.

Sigmund [1] reported the most successful model of sputtering theory that I have known to date, and Andersen and Bay [8] compiled the most complete set of experimental sputtering yields by energetic ions ranging from 0.1 to 100 KeV. However, the situation is less satisfactory for sputtering compound targets of multielements since there exists no universal sputtering theory but only limited experimental data. This phenomenon is usually referred to preferential sputtering; the degree of preferential sputtering depends on projectile energy, mass and target atom binding energy. Compositional changes due to ion bombardment can be significant where growing films are irradiated. Many measurements of preferential sputtering have been reported, and Coburn [9] has compiled data on metal alloys and oxides.

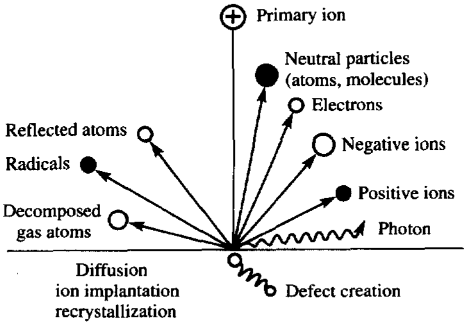


Fig. 2 Schematic diagram of the ion-surface interaction process.

Energy distributions of sputtered particles attain their peak at 1 to 2 eV and extend to about 100 eV. Film properties are largely influenced by the energies of the depositing atoms. In addition, a small percentage of the sputtered particles is ionized, and has a similar energy distribution to the neutral component except for the peak at a higher energy of about 5 to 10 eV [10].

Ion-surface interactions result in the displacement of surface and bulk atoms caused by the collision cascade process which is completed within 10^{-13} sec. Displacement or thermal spikes may occur, and surfaces may undergo crystalline-amorphous transitions. Recoil implantation and cascade mixing create the surface defects, which can enhance diffusion process and accelerate the formation of compounds such as oxide or nitrides [11].

Thin films deposited by magnetron sputtering or ion beam sputtering of a target may be subjected to significant bombardment by primary particles scattered off the target surface onto the growing film. The growing film then contains a percentage of primary particles as well as being subjected to radiation-induced structural changes. On the other hand, the distribution of atoms ejected as a result of ion bombardment from targets does not simply obey a cosine law [12], but tends to concentrate along the direction of so-called focused collisions. Hoffman and Thornton investigated the relationship between the bombardment of the growing film by high energy particles elastically reflected from the target surface and mechanical properties of thin films [13, 14], and concluded that the compressive stress had been caused by an atomic peening effect by the argon atoms reflected from the target surface.

It may be said from the above discussion on the ion-surface interaction that the phenomena occurring during thin film growth by sputtering are very complicated [15, 16]. In order to understand the phenomena, it is necessary to study the parameters for thin film deposition more systematically from the following viewpoint: The influence of kinetic energy of sputtered particles on thin film properties. For this purpose, precise measurements of energy distribution of sputtered ions are first established and then applied to practical SiO_2 and Ni-Si-B films.

In this review, the thin film process of sputter deposition is described on the basis of the effects of kinetic energy of sputtered particles and ion bombardment during deposition on thin film properties.

4. Characterization of Sputtered Particles

In sputter deposition at high gas pressure, film properties are largely influenced by the energies of the depositing atoms. In conventional evaporation, the maximum energy is only ~0.1 eV, whereas sputtered particle energy distributions peak at 1 to 2 eV and extend to about 100 eV [17]. A small percentage of the sputtered particles is ionized and has a similar energy distribution to the neutral component but peaking at a slightly higher energy [10]. However, it is well recognized that the kinetic energy of sputtered ions plays an important role in adding chemical activity to the growing film, which results in changes of the crystallinity, orientation, morphology and adhesion to the substrate [18].

Measurements of the energy distribution (ED) of sputtered ions were performed with an ultra high vacuum - secondary ion mass spectrometry (UHV-SIMS) apparatus. Details were described previously [19]. The energy and mass of the secondary ions were analyzed with an electrostatic ion energy analyzer (IEA) and a quadrupole mass analyzer, respectively. The absolute energy of the IEA was about 2.0 eV. The absolute energy of the IEA was calibrated by Al^+ thermal ions. The sample in the SIMS study was a mirror-polished B-doped Si(111) with a resistivity of 1-2 Ωcm, which was also used as a substrate for thin film deposition. The sample was cleaned by Ar ion sputtering after being introduced into the chamber.

The obtained EDs were characterized by the following parameters:

(1) Most probable energy E_m; (2) full width at half-maximum (FWHM); (3) tail factor N; (4) average kinetic energy E_a. In the usual cases, the N value is defined by the slope of the distribution in the high energy region. In the present study, however, the intensity of the secondary ions in the higher energy region was fairly low. In order to avoid an inaccurate definition of N, the following procedure correction was performed.

We made an assumption that each distribution obeyed Thompson's theory [20] with an N value as a fitting parameter. That is,

$$f(E) = CE \,/\, (E + E_b)^{N+1} \cdots\cdots \quad (1)$$

where E is the energy of sputtered ions, E_b is the surface binding energy and C is a constant. $N = 2$ gives Thompson's theory. The N value was defined by fitting this function to the observed distribution. The N value represents an approximate measure of the distribution function and the E_m value directly associated with the surface binding energy as shown by Thompson's theory.

Although many theoretical models have been proposed to describe the ionization phenomena of sputtered particles, a unified explanation cannot be given at present. The present author [21-24] has established the methods of simultaneous determination of sputtering yield and the ionization probability for thin film targets with known thickness and composition. As a result, the sputtering yield of metals under O_2^+ bombardment was inversely proportional to the so-called energy transfer factor in the classical head-on collision model and a linear relationship was found between the ionization potential and a modified degree of ionization which can be expressed by the ionization potential and the sputtered O_2^+ current.

The essential features of the transport of sputtered particles from a target to a substrate during sputter deposition were studied by calculation using the Monte Carlo technique [25, 26]. The study takes into consideration the change in momentum as well as the kinetic energy loss of sputtered particles in their collisions with ambient gas molecules, to gain an understanding of the effects of these factors and of the number of sputtered particles arriving at a substrate on the mechanism of growth of a thin film by sputter deposition. Some theoretical predictions using the above calculation were made for several selected conditions of sputter deposition.

Fig. 3 shows a typical example of the Monte Carlo calculation of the trajectories of 50 particles during high and low pressure sputter deposition, for easy understanding of the entire transfer process [26].

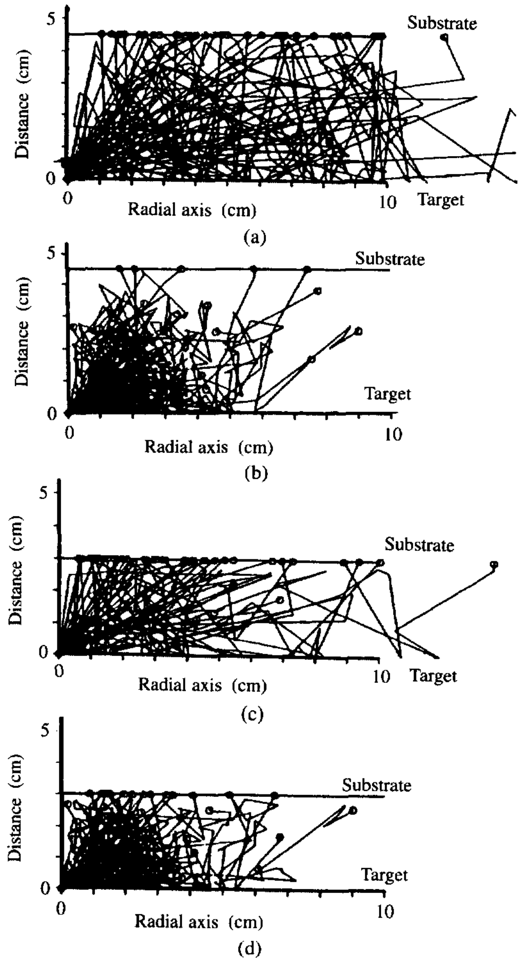


Fig. 3 Trajectories of 50 silver particles ejected into an argon gas ambient from the origin with Thompson's energy and angular distribution at a sputtering voltage of 2 kV and T = 305 K: (a) $P_{Ar} = 6.6 \times 10^{-1}$ Pa., D = 4.5 cm; (b) P_{Ar} = 2 Pa., D = 4.5 cm; (c) $P_{Ar} = 6.6 \times 10^{-1}$ Pa., D = 3.0 cm; (d) P_{Ar} = 2 Pa., D = 3.0 cm.

5. Applications

5.1 SiO_2 Films

SiO_2 films have been recognized to be one of the most important materials in electronic devices and sensors. These films are expected to have high insulative properties even after preparation at low temperature process. In this section, the author tried [27, 28] to elucidate the effects of growth conditions of sputter deposition on the electrical properties of SiO_2 films with a special emphasis on the energy distribution of sputtered particles.

Fig. 4 shows the energy distributions of Si^+, SiO^+, Si_2O^+ and Al^+ thermal ions under oxygen exposure at 5.3×10^{-5} Pa. **Table 1** denotes the most probable energy (E_m) and the average energy (E_a) of secondary ions under different conditions of primary ions (Ar^+, O^+) and targets (Si, SiO^2). The values of E_m in the case of $O^+ \rightarrow Si$ were similar to that of $Ar^+ \rightarrow SiO_2$, while those of $Ar^+ \rightarrow Si^+O_2$ (gas) do not resemble the above cases, that is, the E_m of $Si_mO_n^+$ (m, n = 1, 2, ···) is almost equal to that of Al^+ thermal ions. It is thought that the formation mechanisms of $Si_mO_n^+$ in the case of $Ar^+ \rightarrow Si^+O_2$ (gas) is different from those of $Ar^+ \rightarrow SiO_2$ and $O^+ \rightarrow Si$. It is thought that these molecular ions are created at the surface really as a result of the interaction between the secondary particles and adsorbed atoms. The mechanism of the molecular ion formation mentioned above can be interesting compared to that of chemical sputtering [29, 30].

Radio frequency (RF) magnetron sputtering technique was used for deposition of SiO_x thin films. Hot-pressed SiO_2 and polycrystalline Si targets of 99.9 % purity were bonded to a water-cooled copper backing plate. After evacuation to 1.3×10^{-4} Pa., sputtering gas was introduced to 6.6×10^{-1} Pa. SiO_2 films 50 nm thick were thus deposited at the target-to-substrate distance of 150 mm.

Arrangements in a deposition chamber for

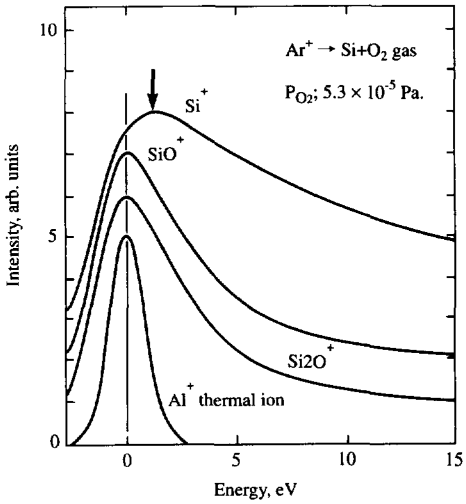


Fig. 4 Changes in ion energy distributions of Si^+, SiO^+ and Si_2^+ from Si under Ar ion bombardment during O_2 exposure to 5.3×10^{-5} Pa.

Table 1 The most probable energy E_m and the average energy E_a of sputtered ions under various conditions. E_a values are shown in parenthesis. * indicates the E_m value equal to Al^+ thermal ions.

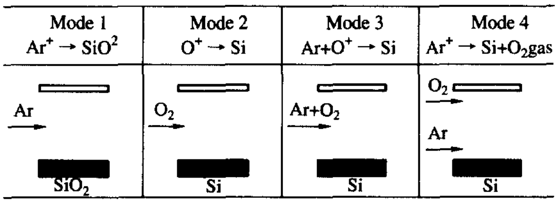
	Si^+	Si^{2+}	SiO^+	Si_2^+	Si_2O^+	Si_2O^{2+}
$Ar^+ \rightarrow Si$	1.1 (11.0)	0.6 (5.2)	—	0.7 (7.0)	—	—
$O^+ \rightarrow Si$	4.9 (16.1)	—	4.2 (7.3)	4.6 (10.9)	3.0 (5.0)	3.3 (4.5)
$Ar^+ \rightarrow SiO^2$	4.2 (17.7)	2.0 (10.7)	2.8 (8.8)	3.2 (12.9)	1.9 (5.2)	1.6 (3.9)
$Ar^+ \rightarrow Si{+}O^2$	1.1 (14.2)	0.6 (7.4)	* (2.4)	0.4 (6.3)	* (1.8)	* (1.0)

thin film preparation were schematically shown in **Table 2**. Modes 1, 2 and 3 were normal methods, but in Mode 4, the experimental arrangement used was specially designed to isolate the target from oxygen gas, maintaining it in an inert gas atmosphere while introducing oxygen to the substrate. As a result, experimental apparatus allowing reactive sputter deposition, in which the chemical reaction occurs at the deposition surface, has been developed.

Results of characterization and electrical properties of SiO_x films are summarized in **Table 3**.

It is interesting to note the results of the measurement of breakdown voltage of the film thus prepared in Modes 1-4. The breakdown voltages E_b of the film by Modes 2 and 4 are high, and those by Modes 1 and 3 are rather low. In order to elucidate the differences in E_b between the films by Modes 2 and 4, the results of *J-E* measurements [31] are depicted in **Fig. 5**, where the *J-E* curve of the thermally grown SiO_2 film is shown for comparison. In the figure, the E_b values of each sample are shown by arrows. Similar E_b values are obtained for Modes 2 and 4, but the changes in *J* with *E* are quite different, that is, *J* in the film by Mode 2 begins to increase at $E = 2.5$ MeV/cm, while *J* in the film by Mode 4 remains unchanged to $E = 5.5$ MeV/cm, and then begins to increase gradually. The *J-E* characteristic of the film by Mode 4 is similar to that of thermally grown SiO_2 films. It can be explained that the current-transport mechanism in the present cases such as high field and low temperature may be a result of the field ionization of the electrons trapped into a conduction band, which is usually referred to as the Poole-Frenkel effect [31]. The trapped electrons in the SiO_2 films prepared by Mode 2 may be induced by high energy bombardment of sputtered particles. On the contrary, the E_a values of the sputtered particles in Mode 4 are low in comparison with Mode 2 and the corresponding trapped electrons in the SiO_2 films thus prepared will be diminished, resulting in the

Table 2 Schematic representation of thin film preparation modes under various conditions.



Mode 1 $\mathrm{Ar}^+ \rightarrow \mathrm{SiO}^2$	Mode 2 $\mathrm{O}^+ \rightarrow \mathrm{Si}$	Mode 3 $\mathrm{Ar{+}O}^+ \rightarrow \mathrm{Si}$	Mode 4 $\mathrm{Ar}^+ \rightarrow \mathrm{Si{+}O_2gas}$
Ar $\mathrm{SiO_2}$	$\mathrm{O_2}$ Si	$\mathrm{Ar{+}O_2}$ Si	$\mathrm{O_2}$ Ar Si

Table 3 Characterization and properties of SiO_x film by Modes 1, 2, 3 and 4.

	Mode 1	Mode 2	Mode 3	Mode 4
Growth rate (Å/min)	96	22	204	63
Density (g/cm^3)	2.00	2.14	1.71	1.90
Refractive index	1.43	1.42	1.47	1.43
Stoichiometry (AES)	$\mathrm{SiO_2}$	$\mathrm{SiO_2}$	$\mathrm{SiO_{1.8}}$	$\mathrm{SiO_2}$
Stoichiometry (IR)	$\mathrm{SiO_2}$	$\mathrm{SiO_2}$	$\mathrm{SiO_2}$	$\mathrm{SiO_2}$
E_b (MV/cm)	4.12	9.9	1.32	9.26

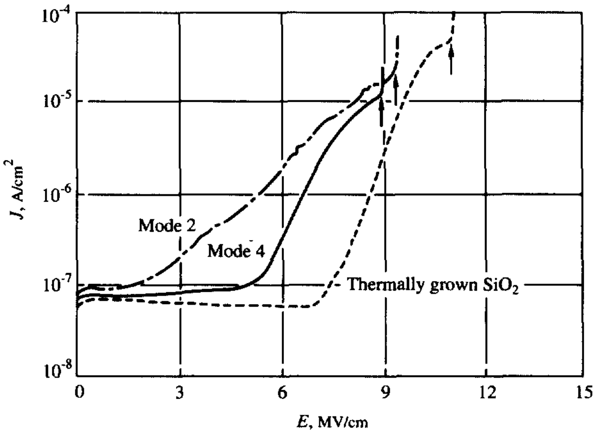


Fig. 5 *J-E* characteristics of Al/$\mathrm{SiO_2}$/Si system for Modes 2 and 4.

same *J-E* characteristic of the thermally grown SiO_2 films (in this case, no defect or vacancy was introduced). The differences in *J-E* plots between the films by Modes 2 and 4 can be reasonably explained in terms of the differences in E_m or E_a of sputtered particles.

Comparison between the data shown in Tables 1 and 3 reveals that the differences in the current-voltage characteristics of SiO_2 films prepared under various sputtering conditions can be reasonably explained in terms of the changes in the most probable energy of the sputtered particles.

5.2 Ni-Si-B Films

The Amorphous Ni-Si-B (NSB) ternary alloy film has been well known as a material with a high tensile strength (2.6 GPa.) and a low temperature coefficient of resistance (TCR, $1\text{x}10^{-6}$/°C) originated from the short mean-free-path (MFP) of electrons in the films. It is also well recognized that the values of MFP are quite sensitive to the microstructure of the films. The kinetic energy of sputtered particles to be deposited may give an important effect on the microstructure of NSB films.

On the other hand, a lot of research has been devoted to determining the film formation mechanism in sputter deposition as well as in other ion-assisted techniques [32-35]. It has become clear that the impact by energetic particles during deposition affects film properties significantly. The effect of the energetic particles reflected from the target surface in sputter deposition has also been studied by several researchers [13, 14, 36]. Until now, there have been few reports which describe the relation between the EDs of the sputtered particles and the film properties.

From this point of view, the effect of the EDs of sputtered particles on film formation, especially on alloy film formation, was determined. Two representative sputtering methods used to prepare ternary alloy films were examined; co-sputtering with multiple elemental targets (co-sputtering) and sputtering with an alloy target (alloy sputtering). First, we measured the EDs of the ions sputtered from elemental nickel, silicon and boron targets and also those from an amorphous Ni-Si-B (NSB) target by ultra high vacuum secondary ion mass spectrometry (SIMS); the former corresponds to co-sputtering and the latter to alloy sputtering. The results were compared with the microstructures of the films prepared by the corresponding methods.

As a result of SIMS analyses, Ni^+ and Ni_2^+ ions were observed from the Ni target, but Ni_2^+ was not detected. On the contrary, various kinds of ions such as Si^{n+}, Si^+, and Si_n^+ (n = 2, 3) were observed from the silicon target. The boron target emitted only B^+, except impurity ions. These EDs were characterized by the parameters described above, and the results are presented in **Table 4**. Most of the curves, except that for Si^+, had lower N values than that given by Thompson's theory.

It is noteworthy that the E_m values of the singly-charged monoatomic ions emitted from the alloy were lower than those from

Table 4 Results of the characterization of the EDs of sputtered ions.

	Ni^+	Si^+	B^+
E_m (eV)	1.9 (1.5)	2.7 (1.9)	2.5 (1.5)
FWHM (eV)	7.1 (6.9)	10.7 (9.8)	13.2 (8.6)
N	1.6 (1.7)	2.0 (1.6)	1.4 (1.3)
E_a (eV)	17.1 (11.3)	15.5 (15.4)	18.4 (14.3)

the elemental targets. The lowering of the E_m values resulted in a decrease in the E_a values of the monoatomic ions. The present results showed that the E_b values of the atoms in the amorphous alloy target were reduced in comparison with those in the elemental targets.

NSB films were prepared by using the sputtering methods corresponding to the energy distribution measurement. An $Ni_{67}Si_{21}B_{13}$ target was used in alloy sputtering. An NaCl single crystal was used as a substrate, on which almost 50 nm thick films were deposited. Also in this case, the compositions of the films were determined by AES and were found to be almost the same, *i.e.* $Ni_{69}Si_{18}B_{13}$ by co-sputtering and $Ni_{69}Si_{21}B_{10}$ by alloy sputtering.

Structures of the NSB films were examined by means of transmission electron microscopy (TEM). The transmission electron diffraction analysis revealed that both of the films were amorphous. **Fig. 6** shows TEM images of the films. Obviously, the two films displayed different images. The film prepared by co-sputtering was relatively uniform in low contrast. There are domains about 20 nm in diameter whose envelop is seen clearly. On the contrary, the film prepared by alloy sputtering exhibited rather a strong contrast and the diameter of the domains ranged from 5 to 20 nm.

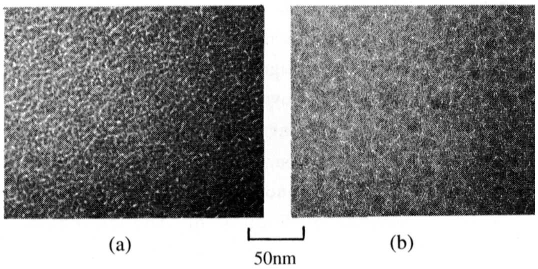


Fig. 6 Transmission electron micrographs of the NBS films prepared by the different deposition techniques: (a) alloy sputtering; (b) co-sputtering.

The contrast could be attributed to mass thickness fluctuation in the film. There are various candidates for the mass thickness fluctuation: Fluctuation in geometrical thickness and that in composition. As a result, it is recognized that the film deposited by co-sputtering has a more uniform mass thickness than the film deposited by alloy sputtering. Two representative sputter deposition methods used to prepare ternary alloy films were examined.

Although it is still difficult to compare the difference in film morphology directly with the difference in the EDs of sputtered ions, the present results suggested that, in co-sputtering, the film grows under particle bombardment with higher energy than in alloy-sputtering. It is also suggested that the difference in the energy of the depositing particles affects their surface mobilities; in film deposition by co-sputtering, particles to be deposited would have a higher surface mobility owing to their higher kinetic energy, which might result in uniform film formation in co-sputtering. The difference in mass spectra, *i.e.* existence or absence of molecular ions such as $NiSi^+$ and NiB^+, might also affect the film structure. Of course, uncertainty still exists concerning the modification of EDs through the transport process. However, the difference in the EDs at the initial stage would affect the final EDs. Further investigation will be necessary to clarify the deposition mechanism.

6. Conclusions

The kinetic energy of depositing the particles, formed by sputtering process in a growing film, strongly influences the structure and property of the resulting films. The electrical properties are particularly sensitive to the film nanostructure affected by the kinetic energy of the sputtered particles. Nowadays, it has become possible to control the film microstructure by the aid of the kinetic energies of both sputtered particles to be deposited and reflected particles which bombard the growing films. Practical applications of high quality electrical films presented in this review are really proofs of the sophisticated thin film technology of sputtering. Further progress of thin film technology must be considered in detail to explore advanced thin film materials science and to ensure the field reliability of future products.

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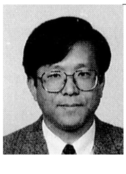

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