ECR イオンビームスパッタ法によって作成された Fe-B-Nb-Nd 磁性金属ガラスのナノインデンテーションによる機械的特性の研究

Nanoindentation study of Fe-B-Nb-Nd soft magnetic metallic glass thin film prepared by ECR ion beam sputtering

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This paper presents some mechanical properties of Fe-B-Nb-Nd based soft magnetic metallic glass thin film that was investigated by nanoindentation testing using a Berkovich indenter with maximum loads of 5 mN and 10 mN. A specimen of the glassy thin film was deposited by electron cyclotron resonance (ECR) ion beam sputtering under 3×10^{-3} Pa of argon inert gas. In order to avoid unexpected effect of an underlying substrate to measured results such as hardness of substrate, thick film specimen of over 2 μ m was prepared for the nanoindentation testing. Hardness of the glassy film as a function of penetration depth and a composite modulus as a byproduct derived from an analysis of the load – displacement curve showed a cause of the indentation size effect. In addition, other factors affecting nanoindentation test data are discussed.

Key Words: nanoindentation, metallic glass, ECR

1. Introduction

Nanoindentation testing is well known as a technique for probing principally mechanical properties of a thin film such as a hardness and composite elastic modulus [1-3]. In nanoindentation testing, these mechanical properties are indirectly calculated to rely on a penetration depth beneath the specimen surface is recognized as the load is applied on the indenter and after removed out. Hardness shows strong dependence as a function of the penetration depth, that is the hardness decreases as the depth increases. This phenomenon is so-called an indentation size effect (ISE). Many experimental and theoretical efforts have been elucidating the ISE, but it does not actually comprehend even taking into account geometrically necessary dislocation, pile-up effect, surface roughness, ratio between energy of newly created surface and plastic strain energy dissipation [1, 4-9]. In addition, several other factors affecting nanoindentation test results are thermal drift, initial penetration depth, instrument compliance, indenter geometry, piling up and sinking in, and residual stresses [1]. The excellent properties of a family of (FeNb)M metallic glass materials in which M are Nd, Y, Gd, or Tb such as excellent magnetism and high glass forming ability have been reported [10,11]. However, there is no attention on these materials as a thin film form than bulk, ribbon, or powder form indeed. Especially, there is till no any report of investigation of the mechanical properties of FeNbNd metallic glass thin film.

In this paper, the hardness and composite elastic modulus of the FeNbNd metallic glass thin film was examined by the nanoindentation testing method. The influence of several external effects to the nanoindentation test data is also discussed.

film were prepared in a chamber at a 3×10^{-3} Pa vacuum pressure of the electron cyclotron resonant (ECR) ion-beam sputtering system (EIS-220, Elionix Co., Ltd.). In this study, (100) oriented silicon wafers with dimensions of 20 mm $\times$ 20 mm were used as substrates for depositing the metallic glass. The silicon wafers were carefully treated using the standard RCA cleaning set, and then etched a very thin natural silicon dioxide of the surface by immersing in HF(1%):H₂O (deionized water) solution. The cleaned wafers were put into the ECR ion-beam sputtering chamber that was subsequently evacuated to a based vacuum pressure of 10^{-4} Pa. Pure argon gas (purity of 99.9999 %) was introduced into the chamber with a flow of 1.54 sccm for exciting plasma occurrence at the head of the ion gun. Then, the argon gas flow was adjusted to the optimized condition of 0.6 sccm for maintaining a vacuum pressure of 3×10^{-3} Pa during depositing process. The sputtering conditions for preparing the specimen of the FeNbNd soft magnetic metallic glass with a high supercooled liquid region of 96 K that was used for the nanoindentation testing are summarized in Table 1.

Table 1. Sputtering parameters for preparing specimens using ECR ion-beam sputtering method.

Sputtering parameters	Value
Acceleration voltage (V)	2400
Microwave power (W)	100
Ion emission (mA)	17.5
Pre-sputtering pressure (Pa)	$< 1 \times 10^{-4}$
Sputtering pressure (Pa)	3×10^{-3}
Argon gas flow (sccm)	0.6
Sample holder's temperature (°C)	30
Sputtering rate (nm/h)	~ 154

2. Experimental procedure

The specimens of (Fe_{0.72}B_{0.24}Nb_{0.04})_{93.7}Nd_{6.3} metallic glass

The specimen with a 2 μ m thick was taken into account to

avoid effect of substrate to nanoindentation testing data when the tip of an indenter was penetrating into the specimen surface. The ultra micro-indentation system (UMIS 2000, CSIRO, Australia) equipped with a sharp triangular diamond indenter (Berkovich indenter) was used to examine a hardness and composite elastic modulus in which it includes both a value of film and that of the Berkovich indenter with different maximum loads to be 5 mN and 10 mN.

A typical example of a loading – unloading curve and schematic of specimen surface after removed an indenter in indentation measurement together with designated parameters are shown in Fig. 1.

Fig. 1. A typical example of loading – unloading curve (a) and schematic of specimen surface after removed an indenter (b). h_r is the displacement of the residual impression, h_e is the elastic displacement during unloading, h_a is the distance from the specimen surface to the edge contact at full load, for the Berkovich indenter $\varepsilon = 0.75$, h_p is the vertical distance along where the indenter penetrated specimen at full load [1, 2].

3. Results and discussion

A loading – unloading hysteresis curve illustrates an oppressive and recovery behavior of the sputter-deposited FeBNbNd metallic glass film during and after withdrawing the indenter from the surface as well as hardness as a function of the penetration depth as shown in Fig. 2. The hardness at adjacent free-surface of metallic glass instantly decreased with increasing penetration depth and then gradually decreased to the lowest value at a maximum depth under $P_{\max} = 5$ mN and 10 mN. This phenomenon refers to the indentation size effect (ISE), i.e. the hardness decreases as penetration depth increases [6]. The ISE is explained by the term of geometrically necessary dislocations that was reported by Nix and Gao for crystalline material, however, for metallic glass there is no dislocation mediated deformation [7, 8]. The ISE of the hardness for metallic glass can be assigned pile-up effect because after taken into account the pile-up effect for both ribbon and bulk metallic glass, the results of nanoindentation did not show such effect [7]. In addition, according to Li [9] the strain softening and friction between the specimen and the indenter facets can take into account for the ISE during doing nanoindentation process of metallic glass. In fact, there are further complicated factors contributing to the ISE should be taken account of hardness analysis of material such as residual stress and blunt sharp tip.

The equations of the hardness and composite elastic modulus are given by [1].

$$H = \frac{P_{\max}}{24.5h_p^2} \quad (1)$$

$$\text{and} \quad \frac{1}{E^*} = \frac{1-\nu_m^2}{E_m} + \frac{1-\nu_i^2}{E_i} \quad (2)$$

where ν_m and E_m are Poisson's ratio and elastic modulus for the material and ν_i and E_i are the same quantities for the Berkovich indenter in this case. For an indenter made by diamond, $E_i = 1141$ GPa and $\nu_i = 0.07$ [3].

In this study, the hardness H was determined about 16.5 GPa $\pm$ 1 GPa and the composite elastic modulus E^* was 138 GPa $\pm$ 10 GPa. Using the equation (2) with the given parameters for the diamond indenter and E^* , we can easily calculate the $E_m/(1-\nu_m^2)$ value to be 156 GPa $\pm$ 11 GPa.

Fig. 2. A loading – unloading curves and hardness values as a function of penetration depth of the FeBNbNd metallic glass film were obtained by performing nanoindentation testing using the Berkovich indenter applied maximum loads of 5 mN and 10 mN.

4. Conclusion

We have produced the FeBNbNd soft magnetic metallic glass thin film with a high glass-forming ability by using electron cyclotron resonant ion-beam sputtering method. The hardness and elastic modulus of the metallic glass film were analyzed using nanoindentation method. It is found that the hardness and the composite elastic modulus of the film are 16.5 GPa $\pm$ 1 GPa and 138 GPa $\pm$ 10 GPa, respectively, through the nanoindentation technique.

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