CHARACTERISTICS OF FeBNbNd MAGNETIC METALLIC GLASS AND ITS APPLICABILITY TO FREESTANDING MICRO-CANTILEVERS FOR ELECTROMAGNETIC TRANSDUCER

T.A. Phan[1*], S. Lee[2], A. Markino[2], H. Okamoto[3], H. Kuwano[1]
[1]Department of Nanomechanics, Tohoku University, Sendai, Japan
[2]Advanced research center of metallic glass, IMR, Tohoku University, Sendai, Japan
[3]Department of Electronics and Information Systems, Akita Prefectural University, Yurihonjo, Japan
*Presenting Author: tuanpa@nanosys.mech.tohoku.ac.jp

Abstract: This paper describes characteristics of the FeBNbNd magnetic metallic glass thin film (MGTF) as well as a fabrication process of freestanding micro-cantilevers and a planar spiral micro-coil by micro-machining technology. Feature properties of the metallic glass film have been systematically characterized such as structure, composition, thermal behavior, mechanical properties, and magnetic properties. In addition, we developed self-standing micro-cantilevers made of the magnetic metallic glass which can be integrated with the planar spiral micro-coil for vibration energy harvesting based on an electromagnetic transducer.

Keywords: metallic glass, glass-forming ability, cantilever, planar spiral coil

INTRODUCTION

Recently, energy harvesting technologies have been playing an important role for widely developing of low power wireless sensors or self power supply of microelectromechanical systems (MEMS) in the range of few μW to hundreds of μW. These technologies based on piezoelectric, electrostatic, and electromagnetic transduction mechanisms utilizing ambient vibrations have been paid more attention to develop new power supplying devices [1-3], which will be desirably alternated the conventional power sources like batteries with a finite life time. New transduction models and novel materials for energy harvesters have been more and more proposed and developed by researchers over the world. In terms of material, one of them is metallic glass material known as a potential material which has been strongly developing for engineering, MEMS applications and mayhap energy harvesting application due to its unique properties such as high toughness, homogeneous, and excellent functional properties [4-7].

In this study, we have mostly focused on studying FeBNbNd magnetic metallic glass thin film (MGTF) and seeking its application for MEMS and especially for a vibration based micro energy harvester utilizing environmental vibrations at low resonant frequency range. The FeBNbNd metallic glass is regarded as an interested material which features many outstanding properties like wide glass-forming ability, high toughness, and excellent soft magnetic properties [8-9]. Deposition process of the FeBNbNd metallic glass on silicon wafer by electron cyclotron resonance ion-beam sputtering method and its characterizations are described in details. We were first attempted to fabricate both freestanding micro-cantilevers and a planar spiral micro-coil using the well-known micro-machining techniques. These MEMS structures would be considered as a new kind of micro energy harvester using metallic glass material in the near future.

EXPERIMENTAL

Fabrication of metallic glass thin film and characterizations

$(Fe_{0.72}B_{0.24}Nb_{0.04})_{93.7}Nd_{6.3}$ metallic glass film on (100) oriented silicon wafer was achieved by depositing 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.). A $Fe_{72}B_{24}Nb_4$ main sputtering target associated with a specific number of $(Fe_{0.7}B_{0.26}Nb_{0.04})_{80}Nd_{20}$ chips was used for controlling a desired proportion of such elements above. Firstly, 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%):H_2O (deionized water) solution. After fixing these silicon wafers into a sample holder, the sputtering chamber was exhausted to a based vacuum pressure of 10^{-4} Pa and pure argon gas (purity of 99.9999 %) was subsequently introduced into the chamber with a flow rate of 1.54 sccm for exciting plasma occurrence at an ion gun. The argon gas flow rate was then adjusted to 0.6 sccm for maintaining a vacuum pressure of 3×10^{-3} Pa during deposition process. The sample holder was kept at room temperature using water cooled chiller system.

The $(Fe_{0.72}B_{0.24}Nb_{0.04})_{93.7}Nd_{6.3}$ metallic glass films on silicon substrates were ultimately characterized for structure, surface morphology, thermal properties, mechanical properties, magnetic properties [10]. An amorphous structure of the as-deposited thin films was determined by an X-ray diffractometer system (XRD - JEOL-JDX Model 3530, JEOL Ltd.) using Cu-K_α radiation together with transmission electron microscopy (TEM) equipped with electron diffraction. Thermal characteristics were analyzed using differential scanning calorimetry (DSC, STD Q600

(TGA/DSC), TA Instruments Inc.) to determine the glass transition temperature (T_g) and crystallization temperature (T_x) at a heating rate of 20 K/min. Supercooled liquid region (ΔT_x), defined as a temperature region between T_g and T_x, is thus obtained. Mechanical properties of the metallic glass film was determined by using the ultra micro-indentation system (UMIS 2000, CSIRO, Australia) with a Berkovich indenter, this is most useful technique for investigating mechanical properties of thin film without removing the film from the substrate.

Micro-cantilevers and micro-coil fabrication

Figure 1 shows a fabrication process diagram of a micro-cantilever by applying micro-machining techniques. Patterns of the micro-cantilever were firstly designed and exposed on a high resolution emulsion glass plate by a high precision laser system, which are used as masks for a photolithography process. Such masks were transferred into a positive photoresist layers coated on a silicon wafer and then developed to remove completely the exposed resist layer (step 1, 2). The micro-cantilever was fabricated as a sandwich structure where the films were successively deposited in order of Ni/FeBNbNd /Ni layers, in which the nickel material was generally used as a protective material against corrosion (step 3). After depositing Ni/FeBNbNd/Ni layers, lift-off procedure was performed (step 4) and then to release the micro-cantilever, a window in the bottom side of the silicon wafer was created based on a positive photoresist (step 5). Finally, the freestanding micro-cantilever was achieved after being released by ICP-RIE dry etching of silicon and removal of the photoresist layer (step 6).

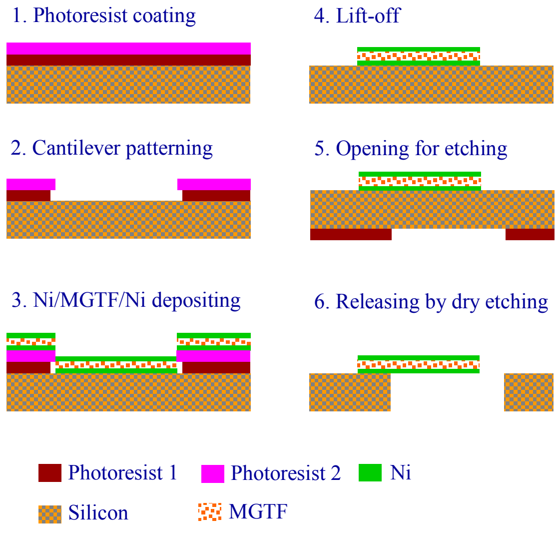

Fig. 1: A fabrication process diagram of a micro-cantilever made of the FeBNbNd metallic glass film with nickel protective layers.

By the way, a mask set of a planar spiral micro-coil was also entirely designed and patterned on the high resolution emulsion glass plates for using a photolithography process. The number of coil turns, width and space between two turns were designed to be 32, 20 µm, and 20 µm, respectively. To fabricate micro-coil, a sandwich structure of $Cu/SiO_2/Cu$ layers was successively deposited using the ECR sputtering system on surface of a silicon wafer with a silicon dioxide layer of 200 nm thick formed by a wet thermal oxidation process. In fact, the first copper layer was used primarily for the micro-coil, which was etched using a wet etching technique. The second copper layer was used for connecting an outer electrode with inner electrode in the center of micro-coil where a window of isolative layer (silicon dioxide) was opened.

RESULTS AND DISCUSSION

Thermal characterization

Thermal stability of the FeBNbNd metallic glass film was characterized using the differential scanning calorimetry analysis in which simultaneous thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) measurements were determined as shown in Fig. 2. The TGA curve illustrates the change of weight of metallic glass as a function of temperature, and on the other hand, the DSC curve shows heat flows associated with thermal transitions in the metallic glass film. Based on DSC curve, the glass transition temperature (T_g) and crystallization temperature (T_x) are readily determined to be 835 K and 952 K, respectively, as shown in Fig. 2 b). The SCLR value is thus obtained to be 117 K. It can be seen that the weight change of the metallic glass is a little bit smaller at lower temperature of T_g, and to seem like constant in the supercooled liquid region as the onset of the TGA curve. On the other hand, the change is instantly raised with an increase of temperature above T_x. Therefore, the thermal stability of the FeBNbNd metallic glass film is distinctly expressed in the SCLR, where the most feature of general metallic glass is to become soft because of its existence in viscous state [4].

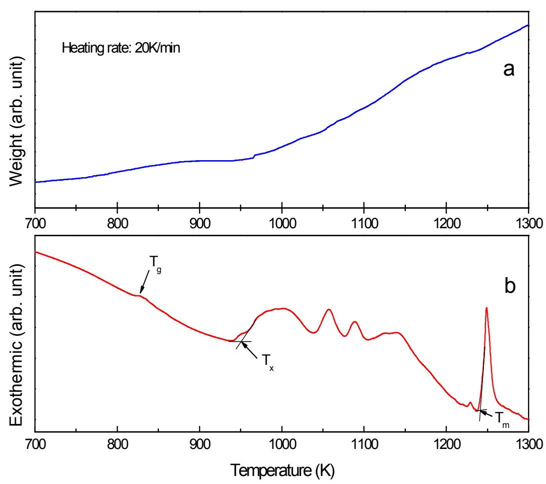

Fig. 2: TGA (a) curve and DSC (b) curve of the 4.5 µm thick FeBNbNd magnetic metallic glass film.

Nanoindentation testing

A loading – unloading hysteresis curve illustrates an oppressive and recovery behavior of the as-deposited metallic glass film during the indenter was penetrating and withdrawing from its surface. Maximum contact pressures of 5 mN and 10 mN were applied to the Berkovich indenter, whereby respective load – displacement curves were recorded and hardness as a function of the penetration depth was thus evaluated for such cases as shown in Fig. 3. The hardness at adjacent free-surface of metallic glass forthwith decreased with increasing penetration depth and then gradually decreased to the lowest value at a maximum depth, this is correct for both P_{max} = 5 mN and 10 mN. This phenomenon refers to the so-called indentation size effect (ISE), i.e. the hardness decreases as penetration depth increases [11-14]. 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 [12]. 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 [11]. In addition, according to Li [13] 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.

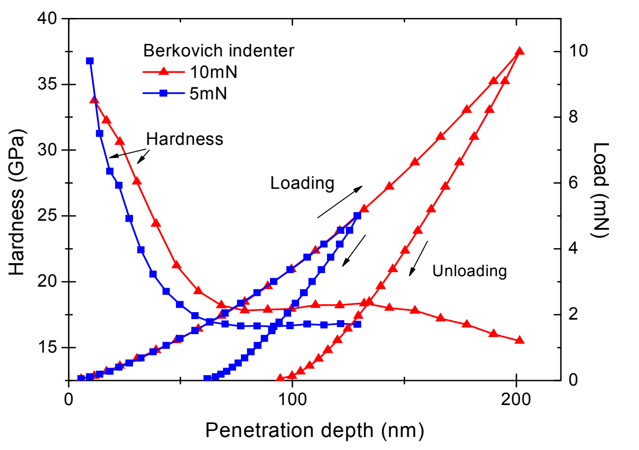

Fig. 3: Loading – unloading curves and respective hardness values as a function of penetration depth of the FeBNbNd metallic glass film were obtained by performing nanoindentation testing using the Berkovich indenter applied maximum loads of 5 mN and 10 mN.

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

$$H = \frac{P_{max}}{24.5 h_p^2} \tag{1}$$

and

$$\frac{1}{E^*} = \frac{1-\nu_m^2}{E_m} + \frac{1-\nu_i^2}{E_i} \tag{2}$$

where ν_m and E_m are Poisson's ratio and elastic modulus for the testing 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 $\nu_i = 0.07$.

In this investigation, the average hardness H was determined about 16.5 GPa ± 1GPa and the composite elastic modulus E^* was 138 GPa ± 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 ± 11GPa.

Freestanding micro-cantilevers MEMS structure

Freestanding micro-cantilevers with different lengths were fabricated by using the micro-machining process as mentioned above as shown in Fig. 4. The purpose for varying the length of the micro-cantilevers is to achieve a certain power output at a wide bandwidth of changing frequencies during the device works. Such micro-cantilevers were in fact deflected in upward direction due to internal stress contained in the as-deposited metallic glass film. Because in practice, it is well known that the film can not be deposited by the sputtering without free stress. Therefore, this trouble would be considered for releasing the internal stress in the metallic glass film before or after release the micro-cantilevers. The micro-cantilevers made of metallic glass are expected to be in possession of high toughness and to reduce resonant frequency to a wide range of environmental frequencies for vibration-based micro energy harvester. In further experiment, a proof mass made of a hard magnetic material such as FePt will be fabricated on the top of free ending of the metallic glass micro-cantilevers.

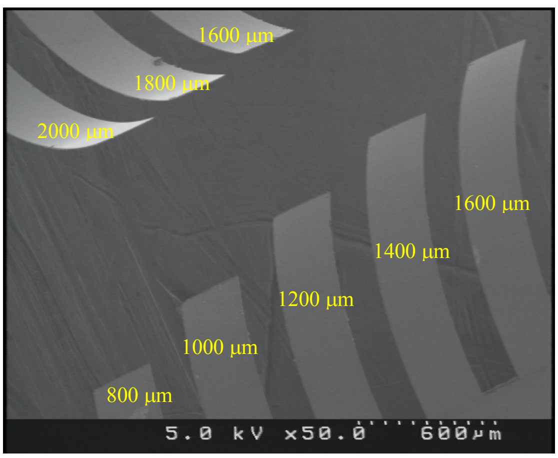

Fig. 4. An SEM image of the magnetic freestanding micro-cantilevers with different lengths (designated in the image for each micro-cantilever).

A planar spiral coil

A completed planar spiral coil was fabricated by using the micro-machining techniques as shown in Fig. 5. The primary part of the copper coil with 2 μm thick was separated completely from the copper line which was used to connect inner electrode and outer electrode by a sputtered silicon dioxide layer with an appropriate thick of several hundreds of nanometers. It was assumed that its inductance could be very small; the practical resistance was measured to be 70 Ω in this case. Furthermore, the further calculations for the planar spiral micro-coil can be seen literatures like Ref. [15]. This coil will be considered to integrate with the fabricated metallic glass micro-cantilevers for a micro electromagnetic generator utilizing vibration source.

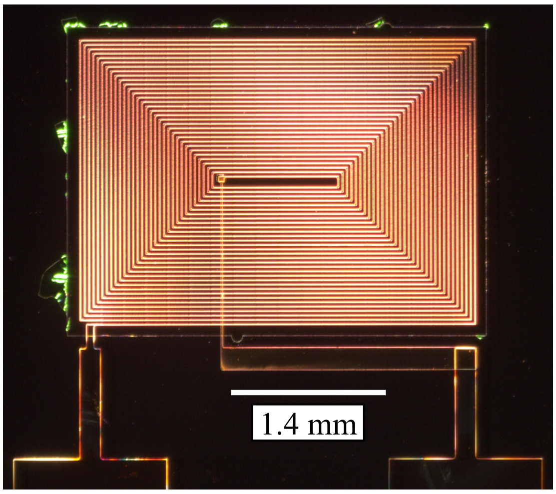

Fig. 5: A completed planar spiral micro-coil to be employed as a passive part of the electromagnetic transducer for vibration energy harvesting.

CONCLUSION

We have developed FeBNbNd metallic glass film using electron cyclotron resonance (ECR) ion-beam sputtering as a candidate material for energy harvesting. Its characteristics were analyzed such as structure, compositions, thermal stability behavior, mechanical properties, and magnetic properties (but some investigations do not present here). The FeBNbNd metallic glass film based micro-cantilevers and planar spiral coil were also attempted to fabricate using micro-machining techniques.

ACKNOWLEDGMENTS

This work was conducted under the project "Research of a nano-energy system creation" (KAKENHI, No. 18GS0203), funded by Japan Society for the Promotion of Science (JSPS), and the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT). One of authors (T.A. Phan) would like to thank the Global Center of Excellence (GCOE) program for financial support.

REFERENCES

[1] Priya S, Inman D 2009 Energy Harvesting Technologies *Springer US, Boston, MA*

[2] Arnold D P 2007 Review of Microscale Magnetic Power Generation *IEEE Trans. Magn.* **43** 3940-3951

[3] Kulkarni S, S Roy, T O'Donnell, S Beeby, J Tudor 2006 Vibration based electromagnetic micropower generator on silicon. *J. Appl. Phys.* **99** 08P511

[4] Hata S, Sakurai J, Shimokohbe A 2005 Thin film metallic glasses as new MEMS materials *18th IEEE MEMS 2005* **62** 479-482

[5] Hilzinger, H. 1985. Applications of metallic glasses in the electronics industry. *IEEE Trans. Magn.* **21** 2020-2025

[6] Hata S, Sato K, Shimokohbe A. 1999 Fabrication of thin film metallic glass and its application to microactuators *Device and Process Technologies for MEMS and Microelectronics* **3892** 97-108

[7] Zhang, G., Y. Liu, and B. Zhang. 2006. Effect of annealing close to Tg on notch fracture toughness of Pd-based thin-film metallic glass for MEMS applications *Scr. Mater.* **54** 897-901

[8] Nowosielski R, Babilas R, Ochin P, Stokłosa Z 2008 Thermal and magnetic properties of selected Fe-based metallic glasses. *Arch. Mater. Sci. Eng.* **30** 13-16

[9] Qiu K Q, Ren Y L 2005. A new Fe-based metallic glass with a larger supercooled liquid region *J. Mater. Sci.* **40** 3305-3307

[10] Phan T A, Lee S M, Makino A, Okamoto H, Kuwano H 2010 Fe-B-Nb-Nd Magnetic Metallic Glass Thin Film for MEMS and NEMS *5th IEEE-NEMS 2010 (Xiamen, China)* (will be published online at http://www.ieee-nems.org/)

[11] Huang Y, Shen J, Sun Y, Sun J 2010 Indentation size effect of hardness of metallic glasses *Mater. Des.* **31** 1563-1566

[12] Nix W, Gao H 1998 Indentation size effects in crystalline materials: a law for strain gradient plasticity *J. Mech. Phys. Solids* **46** 411-425

[13] Li N, Chan K C, Liu L 2008 The indentation size effect in Pd 40 Cu 30 Ni 10 P 20 bulk metallic glass. *J. Phys. D: Appl. Phys.* **41** 155415

[14] Burgess T, Ferry M 2009 Nanoindentation of metallic glasses *Mater. Today* **12** 24-32

[15] Woytasik M, Moulin J, Martincic E, Coutrot A, Dufour-Gergam E 2007 Copper planar microcoils applied to magnetic actuation *Microsyst. Technol.* **14** 951-956