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Development of a FeBNdNb Magnetic Metallic Glass Thin Film and its Application to Magnetic MEMS

著者	PHAN ANH TUAN
号	57
学位授与機関	Tohoku University
学位授与番号	工博第4688号
URL	http://hdl.handle.net/10097/61615

	ファン　アン　チュアン
氏　　　名	Phan Anh Tuan
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指導教員	東北大学教授　桑野　博喜
論文審査委員	主査　東北大学教授　桑野 博喜　東北大学教授　羽根 一博 東北大学教授　江刺 正喜　東北大学教授　牧野 彰宏

論文内容要旨

"Sensor communication society" is a concept proposed by Professor Hiroki Kuwano in 1993. The key to establish this society is micro- and/or nano- electromechanical systems (MEMS/NEMS) in which miniature mechanical components (sensors and actuators) and electrical components (processing circuitry and self-powered supply) are integrated into a single device. Advantages of MEMS/NEMS satisfy the strict requirements of such society such as small size, low power consumption, high sensitivity, self-powered, and low cost. However, the traditional MEMS materials such as crystalline or polycrystalline silicon often suffer from low toughness, brittleness, et al. In such a situation, metallic glasses have collected attention as alternative materials to silicon owing to its superior mechanical properties such as high strength, high elastic limit, high fracture toughness et al. But, in contrast to bulk metallic glass, there are only a few reports of metallic glass thin films, especially magnetic metallic glass thin films, for MEMS applications.

Thus, in this study, I have aimed 1) to fabricate a new magnetic metallic glass thin film to increase variety for MEMS applications and to deepen understandings of its physical properties, 2) to fabricate micro-structure which is applicable to MEMS devices and to evaluate its fracture toughness, and 3) to demonstrate application of the new magnetic metallic glass thin film to a MEMS current sensor.

Chapter I gives introduction of this study including background and purposes.

Chapter II discusses about fabrication of a new magnetic metallic glass thin film. In this study, we have fabricated and characterized a new $Fe_{67.46}B_{22.5}Nd_{6.3}Nb_{3.74}$ (Fe-B-Nd-Nb) magnetic metallic glass thin film. Fe-B-Nd-Nb thin films were deposited on (100) silicon substrates by electron cyclotron resonance (ECR) ion beam sputtering. During deposition, the substrate was kept at room temperature (RT) using a water-cooled substrate stage. Two targets with different compositions ($Fe_{72}B_{24}Nb_4$ and $Fe_{56}B_{20.8}Nd_{20}Nb_{3.2}$) were simultaneously sputtered in high-purity (99.9999%) argon gas of pressure of 5 × 10^{-2} Pa at a power of 100 W and a cathode voltage of -2400 V. The volume ratio of these targets and the deposition power during sputtering were optimized in advance to obtain a composition of $Fe_{65.28}B_{24}Nd_{6.72}Nb_4$, which exhibits the magnetic metallic glass state in bulk form. The obtained thin films were characterized using the following methods. Composition was measured by electron probe microanalysis. Crystallinity was evaluated by X-ray diffractometry (XRD) and transmission electron microscopy (TEM). Thermal properties were examined by differential scanning calorimetry (DSC) between RT and 1373 K at a heating rate of 20 K/min. Magnetic properties were studied by observing the magnetization hysteresis (M-H) loops obtained by applying a magnetic field parallel and perpendicular to the thin-film surface using a physical property measurement system (PPMS).

Figure 1 shows a high-resolution TEM image of the Fe-B-Nd-Nb thin film on a (100) silicon substrate. The random black and white contrast in the region of the thin film indicated its amorphous state. The selected-area electron diffraction (SAED) pattern in the inset shows the halo ring typically observed for amorphous materials and further corroborated the amorphous state of the thin film. The sharp image contrast between the native oxide layer of the silicon substrate and the Fe-B-Nd-Nb thin film indicated that there was no contamination of the thin film from the substrate.

Figure 2 shows a DSC curve of the Fe-B-Nd-Nb thin film. The curve had one broad endothermic peak centered at approximately 900 K and an exothermic peak centered at approximately 960 K. By comparing the DSC curve with that of $Fe_{65.28}B_{24}Nd_{6.72}Nb_4$ metallic glass bulk, the endothermic and exothermic peaks were ascribed to the glass transition and crystallization, respectively. Thus, as shown in Fig. 2, the onset of these peaks gave values of T_g and T_x of 850 and 946 K, respectively. The difference between T_g and T_x also gave an SCLR of 96 K. Interestingly, the SCLR for the Fe-B-Nd-Nb thin film was about twice than that for the $Fe_{65.28}B_{24}Nd_{6.72}Nb_4$ magnetic metallic glass bulk, although the compositions of these materials are almost the same. Also, it should be noted that the value of 96 K is the largest among the reported values for the SCLR of magnetic metallic glass thin films. We consider that a possible reason for the increase in the SCLR upon thin-film fabrication was due to the strain from the substrate acting on the thin film, which may have been controlled by annealing as mentioned above. A study on the relationship between the annealing, the strain, and the SCLR is in progress and will be reported elsewhere.

The value of the in-plane coercivity (H_c) (Figure is not shown here) determined from the M-H loops was 7.5 A/m, which is the smallest among the existing magnetic metallic glass thin films. The value of the H_c for the Fe-B-Nd-Nb thin film was about one-third of that for $Fe_{65.28}B_{24}Nd_{6.72}Nb_4$ magnetic metallic glass bulk.

Chapter III discusses about the fracture toughness of the Fe-B-Nd-Nb thin film in the form of the micro freestanding structure. We adopted the "on-chip" uniaxial tensile testing method proposed by K. Sato, which was successfully used to measure mechanical properties of micro-sturctures made of single crystal silicon, SiO_2, Si_3N_4 and permalloy. We fabricated testing devices by using micro-fabrication techniques, then measured the fracture toughness and observed the fracture behavior of our micron-sized Fe-B-Nd-Nb metallic glass thin film. Figure 3 shows SEM micrographs of a fabricated testing device, a freestanding specimen, and a notch etched by focused ion beam on single edge of the specimen. Averaged fracture toughness and Young's modulus of the Fe-B-Nd-Nb metallic glass thin film measured at room temperature were 6.4 MPa $m^{1/2}$ and 79.4 GPa, respectively. Figure 4 shows a comparison of fracture toughness obtained in this work with a number of materials used in MEMS devices. The measured fracture toughness is the highest compared with other thin films adopted in MEMS, and three times higher than that of silicon as shown in Fig. 4. This verifies significance of the $Fe_{67.46}B_{22.5}Nd_{6.3}Nb_{3.74}$ thin film as a future base material for MEMS.

Chapter IV discusses about demonstration of the Fe-B-Nd-Nb metallic glass thin film to MEMS applications. For this purpose, I developed a new contactless type electric current sensor consisting of the freestanding Fe-B-Nd-Nb magnetic micro-cantilevers as shown in Fig. 5. A principle of the sensor is as follows; the magnetic micro-cantilever externally vibrated at a resonant frequency f_0 is placed close to a conducting wire. Applying current to the wire, the magnetic micro-cantilever feels force due to induced magnetic field around the wire. The strength of the induced magnetic field being exponentially proportional to the applied current, the magnetic force acting on the magnetic micro-cantilever increases and resonant frequency decreases with increase in current.

Firstly we theoretically estimated behavior of the resonant frequency. The relationship between resonance frequency shift and current is expressed by

$$\Delta f = -\frac{(\mu-\mu_0)}{16\pi^3\sqrt{mk}}\left(\frac{1}{a^3}-\frac{1}{(a+t)^3}\right)i^2 \propto i^2$$

Taking the logarithms of both sides of the equation, we have

$$\log(\Delta f) = 2\log(i) + \log(A)$$

where $A = -\frac{(\mu-\mu_0)}{16\pi^3\sqrt{mk}}\left(\frac{1}{a^3}-\frac{1}{(a+t)^3}\right)$, a is an initial distance between the cantilever and the wire, t is the thickness of the micro-cantilever.

The equation implies that the relationship between the resonant frequency shift and the current is linear with a slope of two when the planar magnetic field is considered.

Secondly we empirically observed the behavior of the resonant frequency. Resonance characteristic and sensitivity to the current were evaluated using a Phase Sensitive Detection (PSD) method. As a result, the resonance frequency of 3.85 kHz in the fundamental flexional mode was obtained for the cantilever with 750 μm length × 150 μm width × 3 μm thick. As Fig. 7 shows, the linear relationship was observed when the distance between the cantilever and the current-carrying wire was 0.5 mm and 1.0 mm. The sensitivity of 24.8 Hz/A^2 and 20.7 Hz/A^2 was achieved in each condition.

Chapter V gives conclusion.

In this study, we succeeded to fabricate a new $Fe_{67.46}B_{22.5}Nd_{6.3}Nb_{3.74}$ magnetic metallic glass thin film. Characterization of the thin film showed significant improvement in physical properties such as the SCLR and H_C than those of the bulk counterpart. We also succeeded to fabricate the micro-structure made of the Fe-B-Nb-Nd magnetic metallic glass thin film. The specially designed measurement for micro-structures showed that the Fe-B-Nb-Nd micro-structure has about three times larger fracture toughness than Si. Finally we demonstrated the application of the Fe-B-Nb-Nd thin film to a MEMS current sensor. This study is expected to facilitate research on developing magnetic metallic glass thin films as base materials for MEMS.

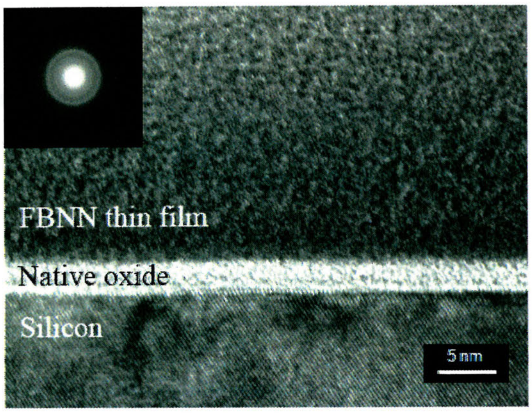

Figure 1: TEM micrograph of a cross section of the as-deposited thin film on Si substrate.

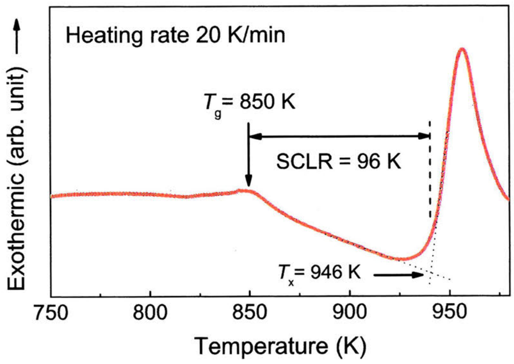

Figure 2: DSC trace of the as-deposited thin film.

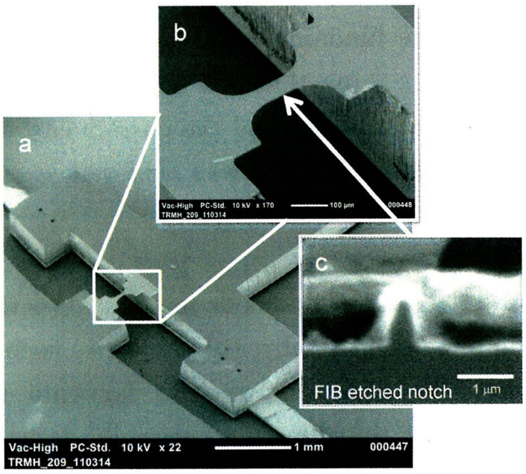

Figure 3: SEM micrographs: (a) testing device and (b, c) enlarged micrographs of the specimen and a FIB etched notch with 1 μm length.

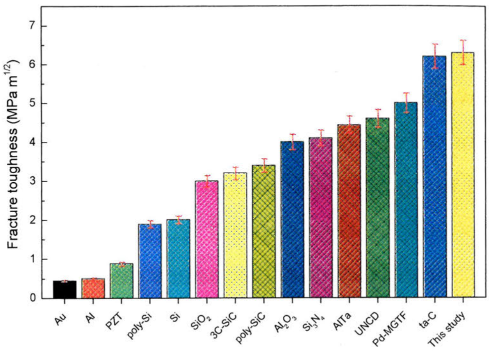

Figure 4: Comparison of the fracture toughness reported for various thin films used for MEMS.

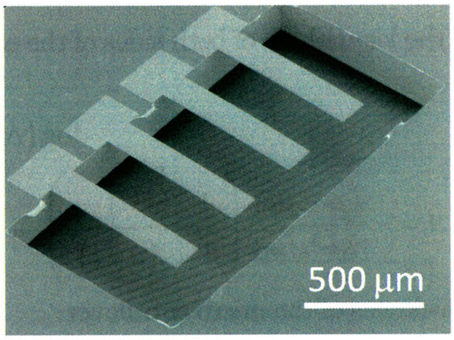

Figure 5: SEM micrograph of freestanding micro-cantilevers made of the Fe-B-Nd-Nb magnetic MGTF: 750 μm length × 150 μm width × 3 μm thick.

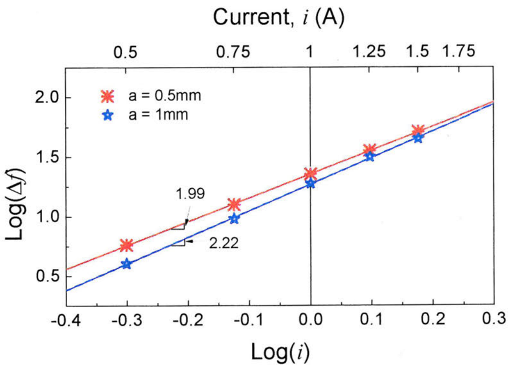

Figure 6: Linear relationship between Log(Δf) and Log(i) of the short cantilever A.

論文審査結果の要旨

MEMS（Micro Electromechanical Systems）はマイクロセンサやマイクロアクチュエータなどとして情報機器等に用いられ新しい装置やシステムを生み出す重要なデバイスである。このMEMSの構造体材料はシリコンであることが多いが，その機械的脆さのため代替材料探索が行われている。有力な代替材料のひとつが金属ガラスであり，破壊靭性が高いため注目されている。しかし金属ガラスに関してはバルクの研究がほとんどで，MEMSで使用する半導体プロセスとの整合性が高い薄膜の研究は報告例が少ない。そのため新規金属ガラス薄膜を開発して材料選択肢を広げ，なおかつ薄膜状態での物性に関する知識を蓄積することが必要である。そこで本論文は，MEMSセンサなどへの応用が期待される新規磁性金属ガラス薄膜の開発と物性評価に取り組み，さらにMEMSへの応用可能性を実証するものであり，全編5章からなる。

第1章では，序論として，本研究の背景，目的および課題と各章における具体的な研究の内容を述べている。

第2章では，新規磁性金属ガラス薄膜の作成手法および物性評価結果について述べている。本論文研究で対象とする新規磁性金属ガラス薄膜の組成（$Fe_{67.46}B_{22.5}Nd_{6.3}Nb_{3.74}$）は，バルク研究で明らかになっている，Fe-B-Nd-Nb四元系における組成と金属ガラスの生成容易さの関係を基準として決定されている。薄膜作成においては汚染物質混入が極めて少ない真空装置を用いており，さらに正確に組成制御された磁性金属ガラス薄膜を高速に堆積できる薄膜作成条件の探索を行っている。得られた薄膜の評価は組成，結晶性，熱力学特性，磁気特性の広範囲に渡っており，これらの評価により明らかになった非晶質性とガラス転移点の存在を総合的に判断して，新規金属ガラス薄膜の作成に成功したと結論付けている。また一般に磁性金属ガラス薄膜ではバルクに比べてガラス状態が安定化し，磁化反転が容易化することが示唆されている。本章で得られる新規磁性金属ガラス薄膜の作成とその物性に関する知見は，今後の材料探索へ寄与する重要な情報である。

第3章では，開発した新規磁性金属ガラス薄膜の破壊靭性評価法と，得られた結果について述べている。破壊靭性は材料の形状やサイズに影響を受けやすい物性であるため，本論文では，新規金属ガラス薄膜からなるマイクロメートルサイズの自立型梁構造を，破壊靭性測定ができる小型測定デバイス上に直接形成している。そしてこの小型測定デバイスの可動部の形状を最適化することで，自立型梁構造部分にかかるわずかな張力の検出を可能としている。得られた新規磁性金属ガラス薄膜の破壊靭性平均値は，シリコンと比べて約3倍，またシリコン以外のMEMSに利用実績のある材料と比べても高い値を示している。このことは新規磁性金属ガラス薄膜がシリコンの代替材料として有望であることを示すだけでなく，金属ガラス薄膜一般のMEMS構造体材料としての適性を示唆しており，今後の材料探索の促進につながる有用な結果である。

第4章では，新規磁性金属ガラス薄膜を構造材料として使用したMEMS電流センサの開発について述べている。この電流センサは新規磁性金属ガラス薄膜を加工したマイクロメートルサイズの自立型片持ち梁構造をしており，電流が形成する磁場を検知して共振周波数が変化するように考案されている。この自立型片持ち梁構造を電流が流れる配線付近に固定しセンサ応答を実験的に検討したところ，電流値の増加に伴って共振周波数変化量が単調に増大するという結果が得られており，定量性を必要とする電流センサへの応用可能性が実証されている。またこの実験結果は理論予想と良く一致している。新規磁性金属ガラス薄膜のMEMSセンサへの応用可能性を実証したことは，今後の実用化への道を切り拓く重

要な知見である。

第5章は結論である。

以上要するに本論文は，シリコンより高い破壊靭性を有する新規磁性金属ガラス薄膜の開発に成功し，MEMSへの応用可能性を実証するものである。加えて本論文研究で得られた金属ガラス薄膜およびそのマイクロ構造体の合成・設計・評価手法に関する知見は，基礎的な材料開発から実用的なデバイス開発にいたる広範な領域で研究指針を提供するものであり，ナノメカニクスおよび情報ナノシステム工学の発展に寄与するところが少なくない。

よって，本論文は博士(工学)の学位論文として合格と認める。