Effect of Aspect Ratio on the bending property of Titanium Fiber Formed by the Compression Shearing Method at Room Temperature

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(Received 7 January 2013; received in revised form 3 April 2013; accepted 20 April 2013)

Abstract
Titanium fiber thin plates were formed by a compression shearing method at room temperature. The effect of the fiber aspect ratio on the microstructure, strength properties, and porosity of the formed samples were examined. As a result, the surface roughness was constant at approximately 5.4um, and the patterned indented surface was homogeneous and isotropic. The porosity of the samples was found to increase from about 32.1 to 39.9% by increasing the aspect ratio of the titanium fiber. The elastic modulus of the samples increased with increasing the aspect ratio of the titanium fiber. In addition, the bending elastic modulus and the bending strength remained constant at approximately 50 GPa and 310 MPa, respectively.

Key words
Titanium, Biomaterials, Plastic Working, Porous Material, Strength Properties, Bending Test, Mechanical Properties, Powder Metallurgy

1. Introduction
Titanium has excellent biocompatibility, strength, and corrosion resistance and so is used as a biomaterial in the field of medicine. Normally, a biomaterial is required to have strength properties equivalent to those of bone, as well as an outstanding affinity to bone. The initial adhesion rate of osteoblast to biomaterial is high in the presence of three-dimensional isotropic irregularities and the surface roughness of titanium is large [1]. In addition, osteoblasts have been shown to exhibit excellent internal growth in titanium biomaterials having a consecutive pore, which diameter is several tens of micrometers [2]. Furthermore, biomaterials used to cover the deficit of skull after skull fractures and prepare the form of bone graft portion after bone grafting are also required to be flexible[3].

However, since rolled pure titanium has a higher elastic modulus and strength than compact bone, there is a need to develop a titanium based material with a reduced elastic modulus and strength. In addition, the surface of rolled pure titanium is smooth, which prevents the adhesion and growth of osteoblasts. One material that may satisfy the above requirements is a porous molded material containing solidified flexible titanium fibers. However, there is a possibility that solidification forming by the conventional powder metallurgy method will prevent reduction in the elastic modulus. Because conventional powder metallurgy method is involve heating, that body become brittle due to oxidation.

Our research group has developed a compression shearing method at room temperature (COSME-RT)[4-10], in which metal powder is simultaneously loaded by a shearing force and a compressive stress in air at room temperature to form a plate without the need for heating [4-10]. Moreover, COSME-RT does not cause embrittlement due to oxidation. Furthermore, a porous, pliable biomaterial that has a three-dimensionally uneven surface is thought to be obtainable by solidification of flexible titanium fiber using COSME-RT.

The goal of the present study is to produce a low-elastic-modulus, low-strength, porous thin plate from titanium fiber using COSME-RT. Titanium fibers with different aspect ratios were formed, and the cross-sectional microstructure, bending strength properties, and porosity of the formed sample were investigated.

2. Sample Preparation
2.1 COSME-RT
COSME-RT is a method of forming a thin metal plate by simultaneously applying a compressive stress and a shearing strain to metal powder or metal fiber.[4-10] A schematic diagram of the COSME-RT process is shown in Fig. 1.

First, pure titanium fibers are placed between a moving plate and a stationary plate in the apparatus. Then, a compressive stress $\sigma_N$ is applied to the moving plate and maintained during the formation process. Next, a shearing load is added to the moving plate, which is displaced in the shearing direction. A thin plate is fabricated in this step.
2.2 Material
In this study, the fiber used for the solidification was fibrous titanium, 99.52 % purity (ASTM grade 1), with a diameter of approximately 20 μm. SEM image of the fibrous titanium are shown in Fig.2. Fibrous titanium was formed by coiled sheet shaving. Fibers with an aspect ratio ($AR = \text{diameter}/\text{height}$) of 25, 100, 250, and 500 were prepared.

![Fig.2 SEM image of the fibrous titanium](image)

2.3 Forming conditions
Specimens were formed using a compression shearing apparatus (Dip, DRD-NNK-001). External view images of specimens are shown in Fig.3. The net amount of titanium fiber was fixed at 1.0 g and the compressive stress $\sigma_N$ was set to 1,000 MPa ($400 \text{ kN}$). The shearing distance was 0.4 mm. The target dimensions of the formed specimens were fixed at approximately 0.6 mm × 40 mm × 10 mm.

![Fig.3 External view images for (a) $AR = 25$, (b) $AR = 100$, (c) $AR = 250$, and (d) $AR = 500$](image)

3. Experimental Procedures
3.1 Surface roughness measurement
In order to investigate the surface properties of the specimens, surface roughness measurement were carried out using a surface shape measurement microscope (KEYENCE, VK-8500). The measurements were carried out in two directions X and Y direction. X-direction is the direction parallel to the longitudinal direction of the specimen, and Y-direction is direction perpendicular to it. Then, arithmetic average roughness $Ra$ was derived from the result.

Fig.4 shows a relationship between aspect ratio and surface roughness. The surface roughness of all specimens was remained constant at approximately 5.4 μm. Also, in all conditions, the surface roughness of Y-direction was same as X-direction. It is considered for isotropic on sample surface, because of the X and Y-direction had same roughness. Since, we observed for sample surface by microscope. Fig.5 (a)-(b) shows that the typical surface images of samples. It can be seen that the surface of the sample keeps fibrous and has same shape for changing AR. Thus, AR was not affect for sample surface.

These mean that the sample formed by COSME-RT is found to have patterned indented surface which was homogeneous and isotropic.

![Fig.4 Relationship between aspect ratio $AR$ and surface roughness](image)

![Fig.5 Images of sample surface](image)

3.2 Cross-sectional observation
In order to investigate the effects of the aspect ratio on the microstructure of the formed specimens, observations of cross sections of the specimens were performed using field-emission scanning electron microscopy (FE-SEM, S-4100, Hitachi High-Technologies Co., Ltd.). Cross-sectional FE-SEM images of the specimens are shown in Fig.6, where it can be seen that the specimen have vacant spaces.

![Fig.6 Cross-sectional SEM images for (a) $AR = 25$, (b) $AR = 100$, (c) $AR = 250$, and (d) $AR = 500$](image)
Fig. 6 subjected image analysis to investigate the porosity. In the result, the specimen of $AR=25$, $100$, $250$ and $500$ have porosity $5.6$, $7.5$, $9.7$, and $15.5\%$. It can be seen that porosity increases as $AR$ increases from $25$ to $500$.

3.3 Bending tests
In order to investigate the strength properties of the specimens, bending tests were carried out using a small tabletop universal testing machine (SHIMADZU, EZ-L-5kN). A test velocity of $0.5$ mm/min was used. Nominal stress and nominal strain of specimens were measured by the load cell and extensometer. Therefore, bending elastic modulus was calculated from true strain measured by strain gauge.

Bending stress-strain curves are shown in Fig. 7, and the curve of rolled titanium (ASTM grade 1) is also shown for comparison. It can be seen that the bending elastic modulus and bending strength of specimen formed by COSME-RT are lower value than the rolled material.

Fig. 8 and Fig. 9 show the dependence of the bending elastic modulus and bending strength on the aspect ratio. Respectively, the values of rolled Ti and compact bone are also shown for comparison. It can be seen that both remained constant at approximately $50$ GPa and $310$ MPa. These values are about $3$ times larger than those for compact bone and about $2$ times smaller than those for rolled titanium. It means all samples have mechanical properties that make it suitable for use as a biomaterial.

3.4 Mercury intrusion test
In order to investigate the porosity more detail of specimens, mercury intrusion tests were performed. Mercury intrusion test is a test that uses the characteristics known as large surface tension of mercury [11]. In order to penetrate the mercury into vacant spaces of the sample, apply pressure to mercury. The porosity and pore diameter of specimens can be determined from the cumulative volume and the applied pressure of mercury.

Fig. 10 shows the relationship between the total injected mercury volume and the applied pressure for different $AR$ values. The curves are seen to become successively higher as $AR$ increases. Fig. 11 shows the total cumulative volume and average pore diameter which calculated values based on these results. It can be seen that the total cumulative volume increases from $109$ to $153$ mm$^3$/g with $AR$. Whereas, the average pore diameter remains constant at approximately approximately $80$ μm.

Fig. 12 shows the dependence of the porosity on $AR$. The porosity is seen to increase from about $32.1$ to $39.9\%$ as $AR$ is increased, although there is evidence of saturation occurring. However, this result is significantly different from that obtained from the cross-sectional SEM observations. Because, the mercury intrusion test is evaluation for three dimensions. On the other hand, the cross-sectional observation is evaluation for two dimensions. As a result, the cross-sectional observation observed vacant spaces less than the actual.
Usually, the elastic bending modulus is decreased with increasing the porosity. However, the porosity was increased with increasing aspect ratio in this study. On the other hand, the bending elastic modulus was stable on all aspect ratio. This phenomenon was related to be the pore diameters. Fig.13 shows that the pore average diameter on each aspect ratio. It can be seen that the all aspect ratio have similar behaviors.

Miura et al. pointed out that the pore diameter and shape of sintered iron were effect to elastic modulus [12-13]. Therefore, bending strengths of all aspect ratio samples were stable. Their samples had large pores about 100µm. This indicates that the large pores became cracks in bending test, and the bending strength had a similar behavior. Since, it is thought that the similar pore diameters of all aspect ratio were effect to stable elastic modulus and the strength in this study.

4. Conclusion
In this study, the Titanium fiber thin plate was developed as a biomaterial using COSME-RT. Therefore, bending properties and porosity of the formed samples were measured. The following results were obtained:

- The surface roughness was constant at approximately 5.4 µm, and the patterned indented surface was homogeneous and isotropic on all aspect ratio.
- The porosity of the samples increased about 32.1 to 39.9 % with fiber aspect ratio.
- The average pore diameter remained the same at approximately 80 µm.
- The bending elastic modulus and bending strength remained constant at approximately 50 GPa and 310 MPa. The value of bending elastic modulus was about 2 times smaller than the rolled titanium (ASTM grade 1).
- Samples of all aspect ratio had the similar pore diameters. This indicates the stable bending elastic modulus on all aspect ratio.

Thus, in the present study we obtained a porous material with mechanical properties that make it suitable for use as a biomaterial.

Nomenclature
COSME-RT
Compression shearing method at room temperature
σN Compression stress
AR Aspect ratio
Ra Arithmetic average roughness [µm]

Acknowledgement
This research was supported by the Program for Fostering Regional Innovation in Nagano, granted by MEXT, Japan.

References


