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Fabrication of Metal Nanohoneycomb Structures and Their Tribological Behavior

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Abstract
Metal nanohoneycomb structures were fabricated by E-beam evaporation and a two-step anodization process in phosphoric acid. Their tribological properties of adhesion and friction were investigated by AFM in relation to the pore size of the nanohoneycomb structures. Variations of the adhesive force are not found with pore size, but formation of the pore greatly reduces the adhesive force compared to the absence of pore structure. The coefficient of friction increased nonlinearly with pore size, due to surface undulation around the pore. Tribological properties do not differ greatly between the original nanohoneycomb structure and the metal nanohoneycomb structure.

Keywords
Nanohoneycomb structure, anodization, adhesion, friction, atomic force microscopy

1. Introduction

As the size of devices shrinks, micro- and nanotribological properties become important because the large surface-area-to-volume ratio causes surface forces such as adhesion and friction to dominate inertial and gravitational forces [1–4]. A comprehensive micro/nanotribological characterization of the scale-dependence of material properties is of great importance in the design of reliable industrial applications, and to provide a bridge between science and engineering on micro/nanoscales. For example, the adhesive force in microcripping systems prevents high precision positioning, causing system failure for moving elements in microsystems. Much re-

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search is therefore aimed at reducing the adhesive force between contact elements in the microsystems using a surface coating or chemical deposition methods. Such methods are, however, temporary, expensive and laborious.

Highly ordered pore array in anodic aluminum oxide (AAO) has been widely studied for its advantages of simple fabrication, short fabrication time and low fabrication cost compared to other nanostructures [5–8]. These AAO structures can be referred to as *nanohoneycomb structures*, because AAO has a honeycomb cell structure with nanometer size pores. Nanohoneycomb structures such as AAO films can be used more widely than honeycomb structures with macroscale cells, with application in the new fields of micro-electro-mechanical systems (MEMS) and nano-electro-mechanical systems (NEMS) devices [9, 10].

AAO as originally developed is ceramic, so that it is nonconductive and brittle. Many studies therefore focus on fabrication of the metal nanohoneycomb structures in order to develop flexible and conductive structures for application in MEMS/NENS devices. The electron-beam lithography method and the electroplating method are widely used to fabricate metal nanohoneycomb structures with regularly arranged pores. These methods are expensive and fabrication is slow. The present study introduces a method for fabricating metal nanohoneycomb structures by anodization in phosphoric acid and E-beam evaporation. The resulting tribological behavior, such as adhesion and friction of metal nanohoneycomb structures, is investigated depending on the pore size, and is compared with the behavior of the original nanohoneycomb structure prior to fabrication.

2. Fabrication of Metal Nanohoneycomb Structures

Metal nanohoneycomb structures are fabricated using an E-beam evaporator, based on a two-step anodization process. Figure 1 shows the fabrication process for metal nanohoneycomb structures. First, the original nanohoneycomb structure is fabricated. Details of the fabrication of this structure by two-step anodization are given in refs. [5–8]. Metal is then evaporated on the nanohoneycomb structure using the E-beam evaporator. After etching aluminum and then alumina, the metal nanohoneycomb structure is complete.

We used phosphoric acid to fabricate the original nanohoneycomb structures. The interpore distance was 500 nm, and the initial pore size was 110 nm. To obtain various pore sizes, widening was performed. Figure 2 shows the five kinds of nanohoneycomb structures with different pore sizes. The pore diameters were 160, 200, 270, 320 and 380 nm, measured by SEM. The thickness of the nanohoneycomb structures was always 10 μm; these are very high aspect ratio structures.

Although we used nickel to fabricate our metal nanohoneycomb structures, any kind of metal can be used. Figure 3 shows the metal nanohoneycomb structures fabricated in this study. The pore diameters were 150, 180, 220, 270 and 330 nm. After the evaporation process, the pore diameters of the metal nanohoneycomb structures were smaller than those of the original nanohoneycomb structures. The thickness of

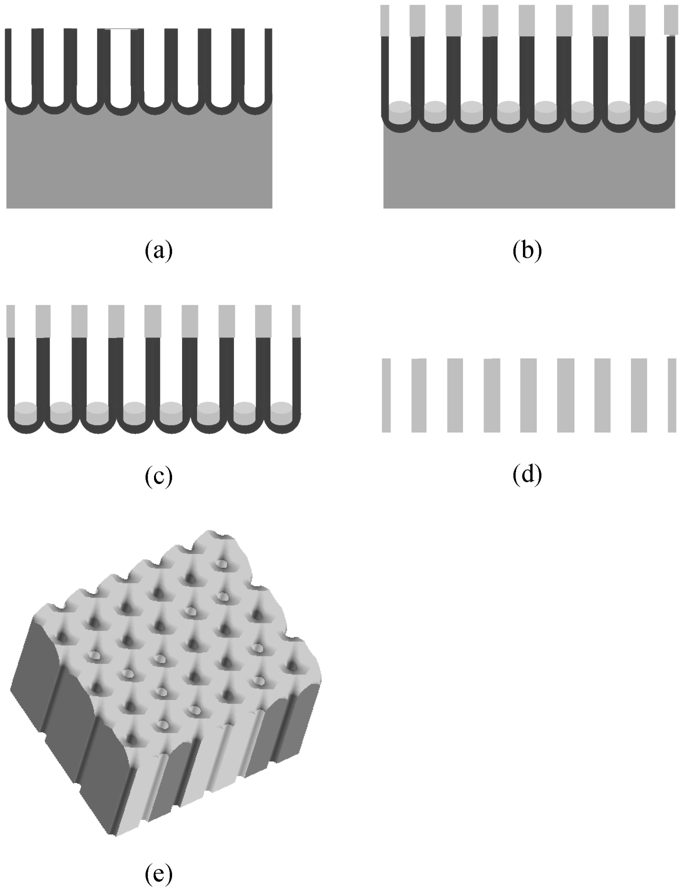


Figure 1. Fabrication process of metal nanohoneycomb structure: (a) Preparation of original nanohoneycomb structures; (b) Metal evaporation; (c) Aluminum etching; (d) Alumina etching; (e) Metal nanohoneycomb.

the metal nanohoneycomb structures was always 300 nm. The thickness can be controlled by the evaporation time but, because the metal particles are deposited on the side wall of the pore and in the pore during evaporation, the pores become smaller and finally close. Figure 4 shows top and cross-section views of metal nanohoneycomb structures having thicknesses 300 and 1000 nm fabricated on identical original nanohoneycomb structures with pore diameter 380 nm. It is clear from Fig. 4(d) and 4(e) that the pores of the metal nanohoneycomb structure with thickness 1000 nm were closed. It is clear that the proposed method is very easy, cheap and fast, but is not well suited to the fabrication of metal nanohoneycomb structures with high aspect ratio. After fabrication of the metal nanohoneycomb structures, the aluminum was etched by $HgCl_2$ at room temperature and the alumina was etched in a mixture of 1.8 wt% chromic acid and 6 wt% phosphoric acid at 65°C, to give the desired metal nanohoneycomb structure.

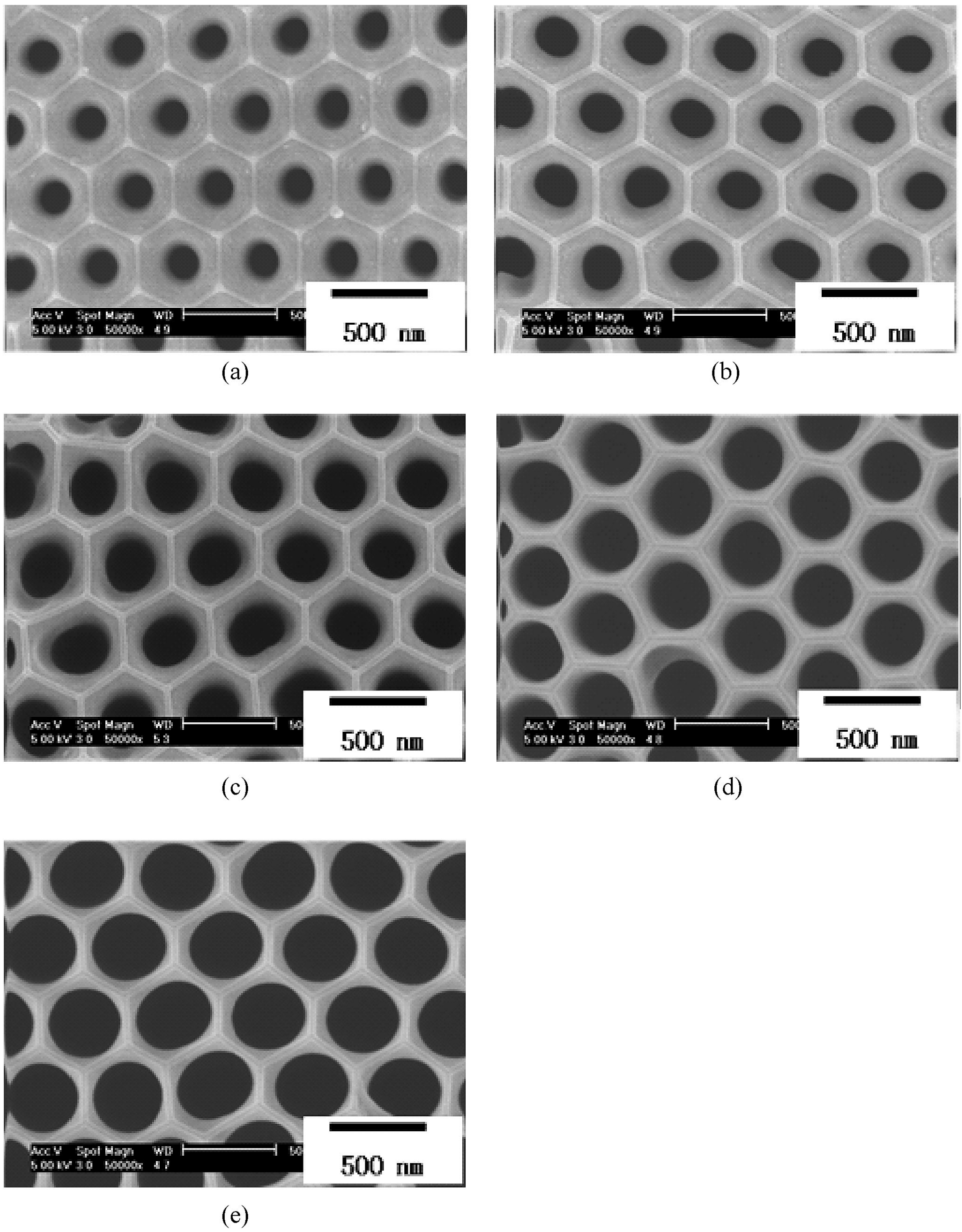
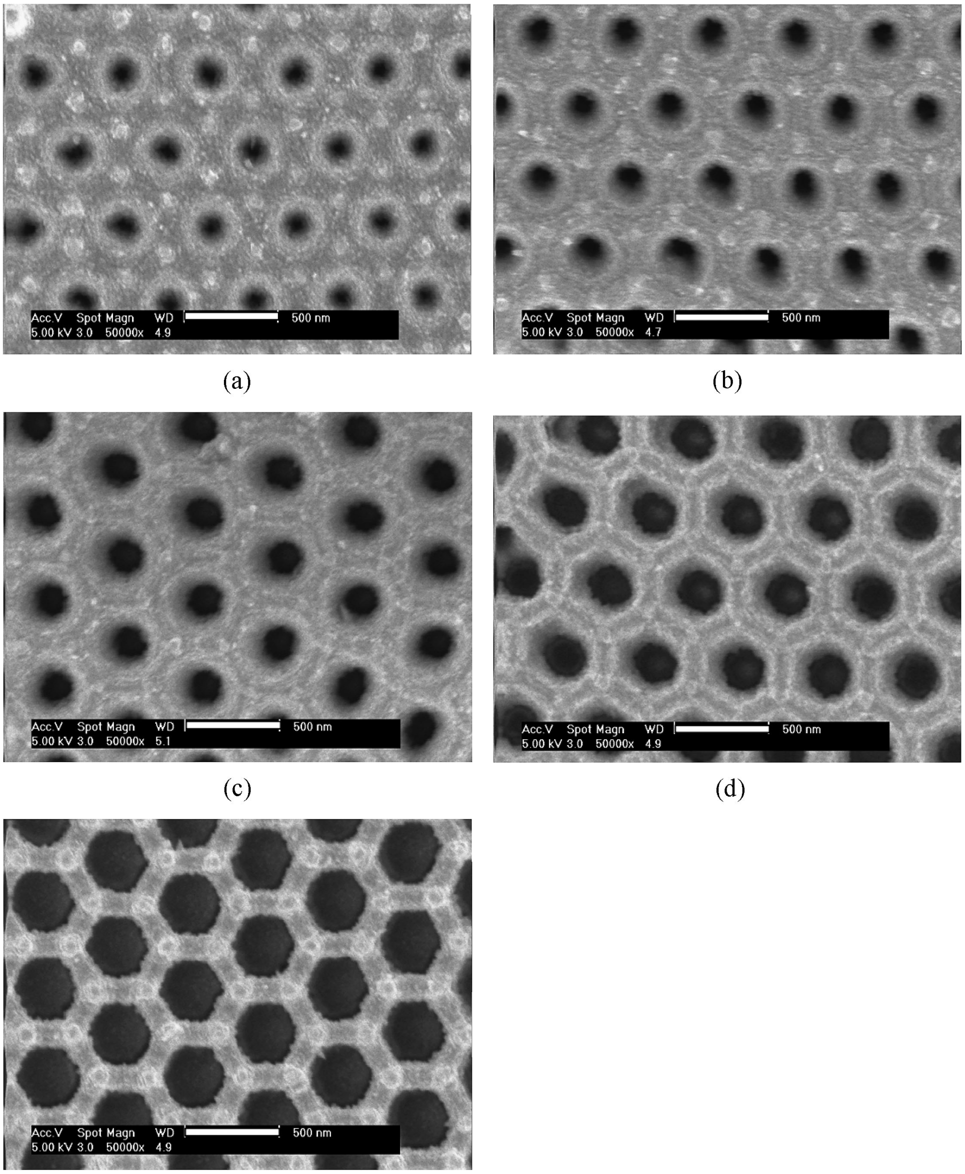


Figure 2. Original nanohoneycomb structures of (a) 157 nm, (b) 200 nm, (c) 272 nm, (d) 316 nm and (e) 382 nm pore size.

3. Experimental Measurements

An AFM (Seiko SPA 400) was used to measure the adhesive force and the frictional force of the nanohoneycomb structures according to their pore size. SiO_2 tips hav-



(a) (b) (c) (d) (e)

Figure 3. Metal nanohoneycomb structures of (a) 150 nm, (b) 180 nm, (c) 220 nm, (d) 270 nm and (e) 330 nm pore size.

ing radii 380, 930 and 2280 nm (fabricated by the Novascan company) were used in adhesive and friction tests. The temperature and humidity were maintained constant during all tests. Alumina and nickel specimens without pores were fabricated as references for the tribological properties of the nanohoneycomb structures.

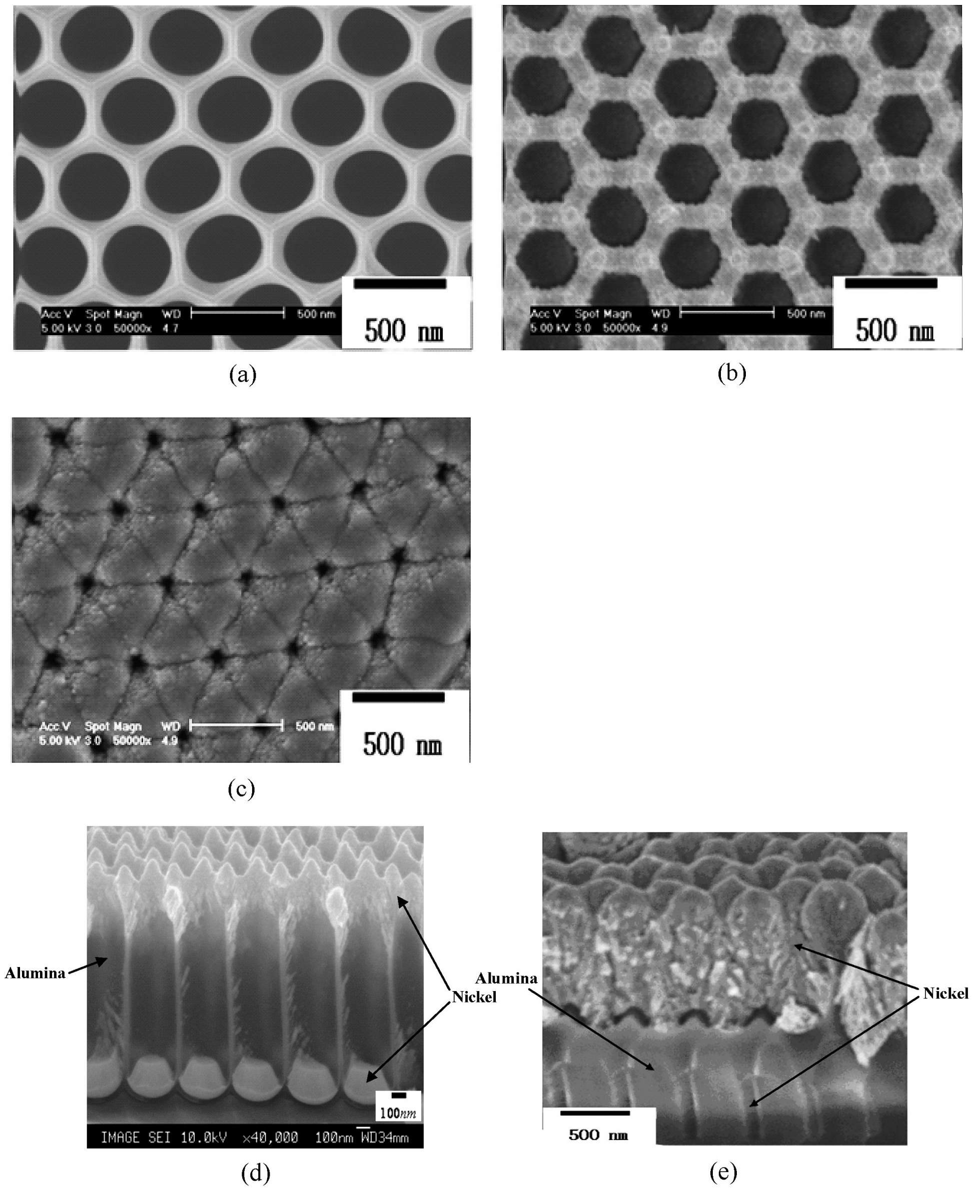


Figure 4. (a) Original nanohoneycomb structure of 380 nm pore size; (b) top view of metal nanohoneycomb structure having 300 nm thickness and (c) 1000 nm thickness; (d) cross-section view of metal nanohoneycomb structure with 300 nm thickness and (e) 1000 nm thickness.

Figure 5 shows the adhesive force of the original nanohoneycomb structures as a function of the pore size for each tip. It confirms that pore formation greatly reduces the adhesive force, though the adhesive force does not vary with the pore size. For the alumina without pores the adhesive force increased from 90 nN to

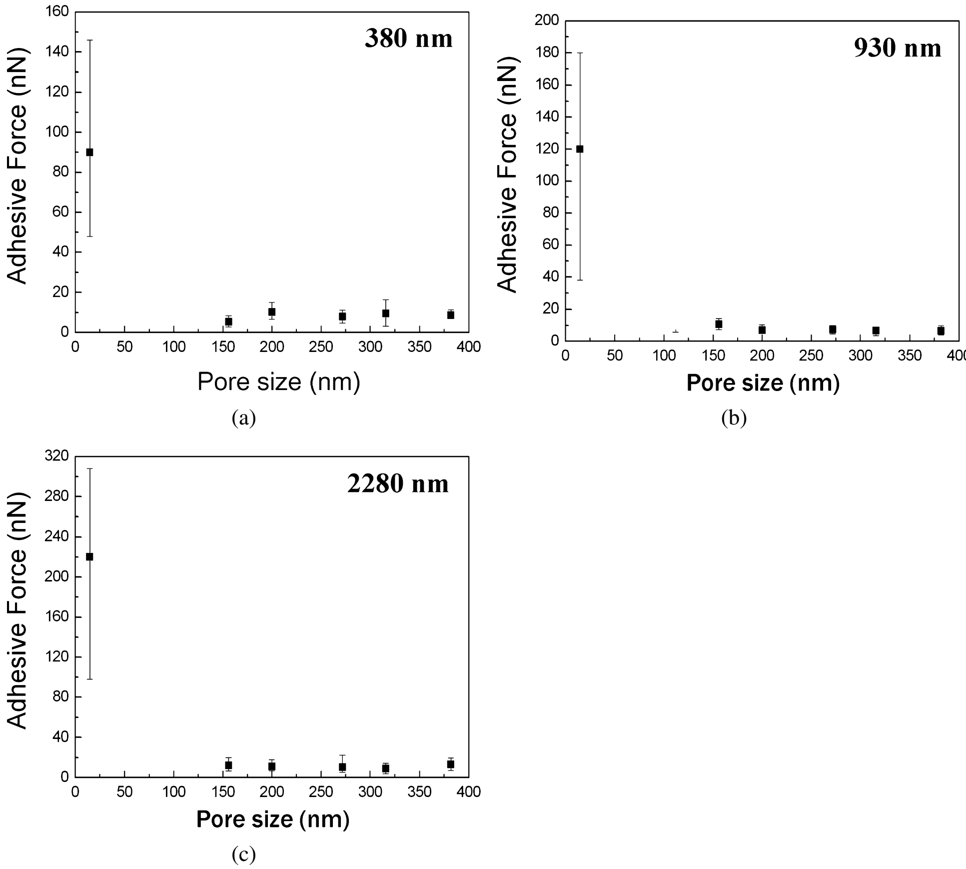


Figure 5. Adhesion results of original nanohoneycomb structures according to the pore size for (a) 380 nm, (b) 930 nm and (c) 2280 nm.

220 nN as the tip size increased, but there were no large changes for the nanohoneycomb structures. Consequently, the nanohoneycomb structures can be used in NEMS and MEMS devices to reduce adhesion problems. In Fig. 6, the frictional coefficient of the original nanohoneycomb structures increases nonlinearly with pore size, and increased with increasing tip size. In general, the frictional coefficient of porous materials decreases with increasing porosity (i.e. pore size) as a result of the decreasing contact area. Figure 4 shows that the top surface of the nanohoneycomb structures fabricated by anodization is not flat. The shape round a pore is approximately a crown shape. It is believed that these undulations round a pore are responsible for the increase in the frictional coefficient of nanohoneycomb structures according to pore size.

Figures 7 and 8 show the adhesion force and the frictional coefficient of the metal nanohoneycomb structures. The adhesive forces were similar to those of the original nanohoneycomb structures. The frictional coefficients of the metal nanohoneycomb structures were smaller than those of the original structures. The tribological prop-

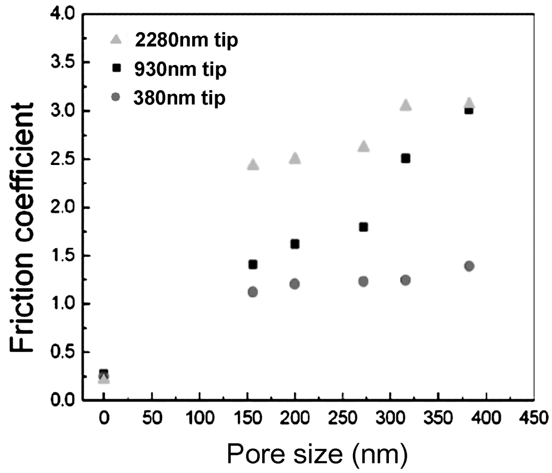


Figure 6. Friction results of the original nanohoneycomb structures according to the pore size.

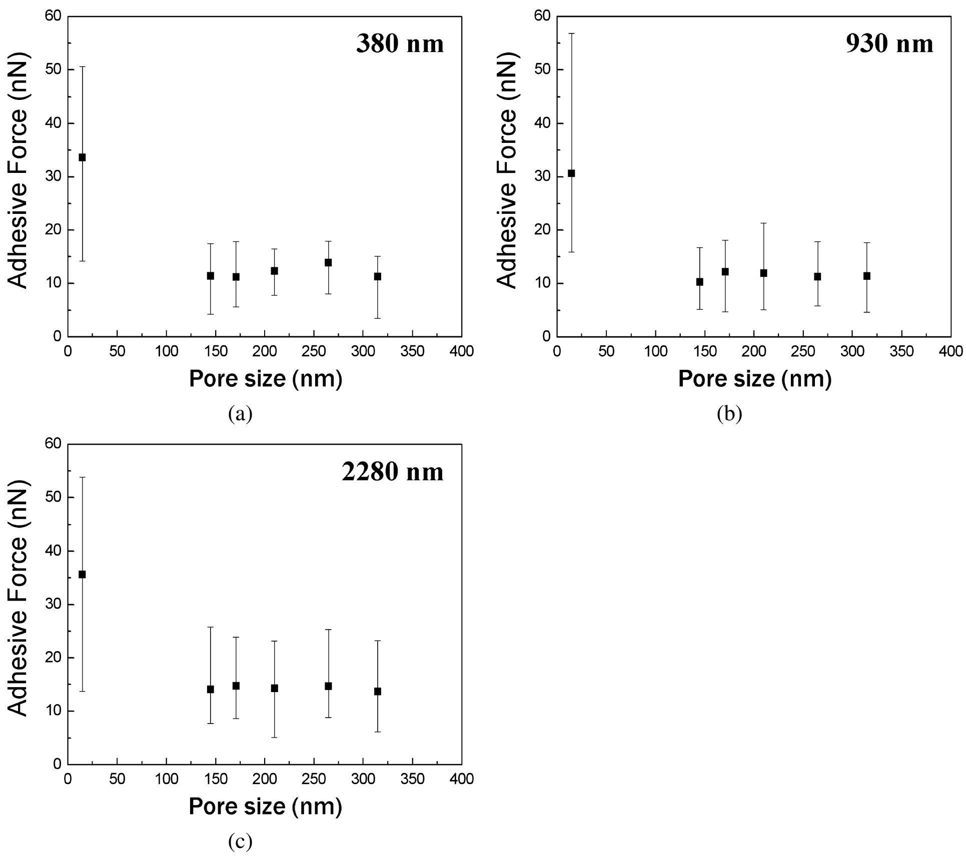


Figure 7. Adhesion results of metal nanohoneycomb structures according to the pore size for (a) 380 nm, (b) 930 nm and (c) 2280 nm.

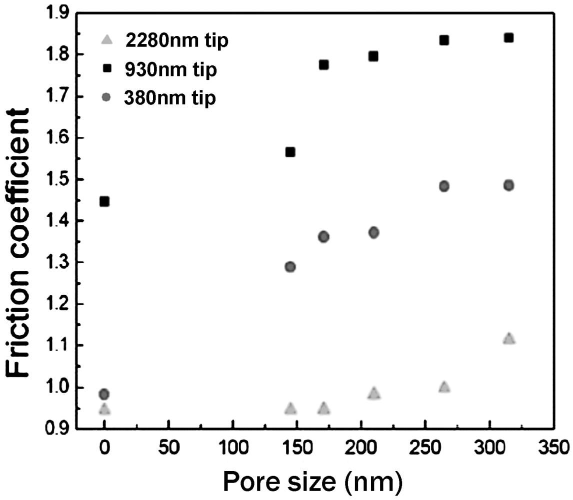


Figure 8. Friction results of metal nanohoneycomb structures according to the pore size.

erties of the metal nanohoneycomb stucture are therefore similar to or better than those of the original nanohoneycomb structure. The original nanohoneycomb structures are, as mentioned, poorly suited for use in NEMS/MEMS devices because of their brittle and nonconductive properties. Since the metal nanohoneycomb structure is more flexible and conductive and has good tribological properties, it makes a better top surface or contacting element in NEMS/MEMS devices.

4. Conclusions

We have verified a simple, cheap and fast method for fabricating metal nanohoneycomb structures. The nanohoneycomb structures greatly reduce the adhesive force. We do not observe large variations depending on the pore size. The frictional coefficient increases nonlinearly with the pore size as a result of the undulation around the pores. The metal nanohoneycomb structures displayed tribological properties similar or superior to the original nanohoneycomb structures. The metal nanohoneycomb structures cannot, however, be fabricated with high aspect ratio, because metal particles are deposited on the side wall of the pores and within the pores.

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