

# HYDROGEN ANNEALING PROCESS TO IMPROVE FRACTURE TOUGHNESS OF SILICON COIL SPRING FOR MECHANICAL ENERGY STORAGE APPLICATION

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## Abstract:

A silicon coil spring is useful to store mechanical energy in microscale, but the practical energy density is limited by the fracture strength of silicon. In this study, hydrogen annealing process was applied to smooth out the surface roughness of the silicon coil springs and improved the fracture toughness. We demonstrated the hydrogen annealing process was effective to increase the fracture toughness of silicon coil springs made by Bosch process. The stored mechanical energy increased 3.5 times in average and 5 times at the maximum by this process.

**Keywords:** silicon coil spring, scallops, scratches, hydrogen annealing, Bosch process, stored mechanical energy

## INTRODUCTION

Coil springs have been used as energy storage mechanisms which are utilized in mechanical watches as for a long time. Takion Co. Ltd., has proposed a silicon coil spring to store mechanical energy harvested by some mechanical motions [1, 2].

Silicon is basically a suitable material for a spring, because it is an ideally elastic material with high fracture strength. It is also easy to process by wafer-level batch process. Silicon hair springs are now used in Swiss watches, while a silicon coil spring for energy storage has not been well investigated. In comparison with secondary batteries, the coil spring has negligible energy discharge for a long period, but its energy density must be largely improved. If the coil spring is made of silicon, the practical energy density is limited by the fracture strength of silicon.

The strength of a single-crystal silicon is known to be very strong intrinsically. However, it is usually decreased by etching damages caused due to the fabrication process such as deep reactive ion etching (D-RIE) and so on. In this study, therefore, we applied hydrogen annealing process to smooth out the surface roughness of the silicon coil springs to improve the fracture toughness, and evaluated the effects.

## DESIGN AND FABRICATION

Silicon coil springs were fabricated on a 525- $\mu\text{m}$ -thick (100) silicon substrate by D-RIE, as illustrated in Fig. 1. One of the fabricated springs in this study is shown in Fig. 2. The dimensions are summarized in Table 1 and Fig. 3. These designs were based on conventional steel coil springs for our supposed application. After the etching, the “scallops” due to Bosch process and “scratches” originated from resist

edge roughness were formed on the silicon surface. Then, these etching damages were recovered by hydrogen annealing.

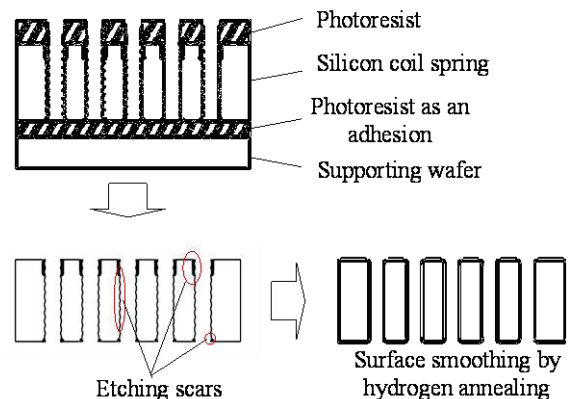


Fig. 1: Fabrication process of silicon coil spring.

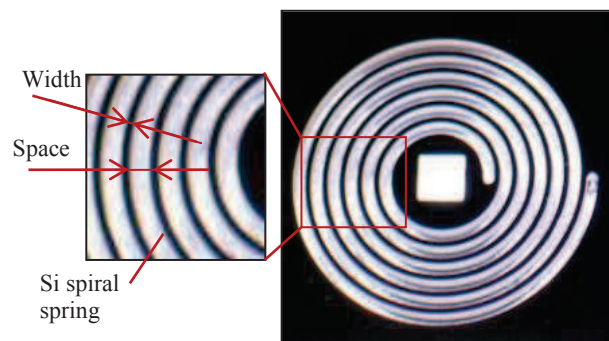


Fig. 2: Optical microscope image of the coil spring (Type A\_R)

Table 1 Dimensions of silicon coil springs.

| Sample name | Width [ $\mu\text{m}$ ] | Space [ $\mu\text{m}$ ] | Total length [mm] |
|-------------|-------------------------|-------------------------|-------------------|
| Type A_R    | 100                     | 100                     | 37.5              |
| Type A_S    | 80                      | 100                     | 41.6              |
| Type A_F    | 120                     | 100                     | 33.6              |
| Type B_R    | 100                     | 100                     | 236.3             |

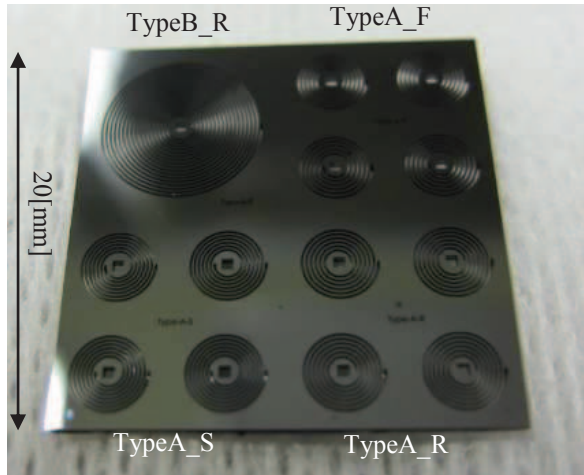


Fig. 3: Prototyped silicon coil springs in this study.

## HYDROGEN ANNEALING

It is known that hydrogen annealing process at 1000°C or higher can reduce the surface roughness of silicon due to reflow effect [3]. This process was applied to the silicon coil springs. The annealing condition is shown in Table 2, which was optimized by Kanamori *et al.* [4].

Fig. 4 shows the scanning electron micrographs of the silicon coil spring without the hydrogen annealing process. The scratches are clearly found near the corner. By contrast, these could be almost removed by the hydrogen annealing, as shown in Fig. 5. This smoothing effect was expected to decrease stress concentration points on the surface, which would result in increase of storage energy density.

Table 2 Annealing Condition

| Pressure[kPa] | Temperature[°C] | Time[min] |
|---------------|-----------------|-----------|
| 50            | 1200            | 30        |

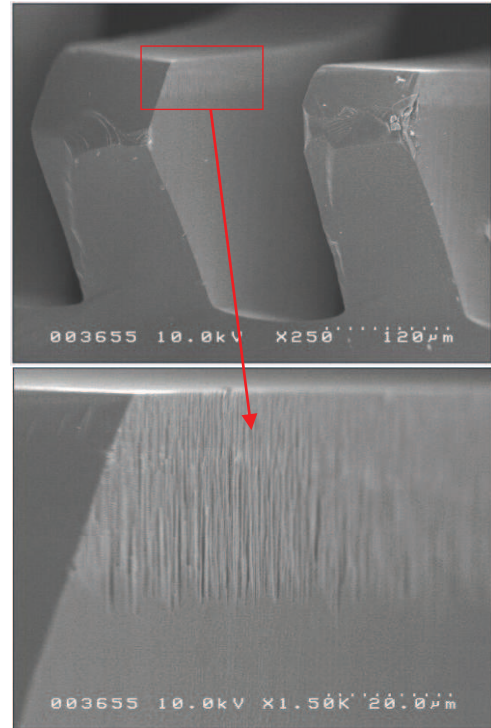


Fig. 4: SEM photograph of DRIE sidewall without annealing.

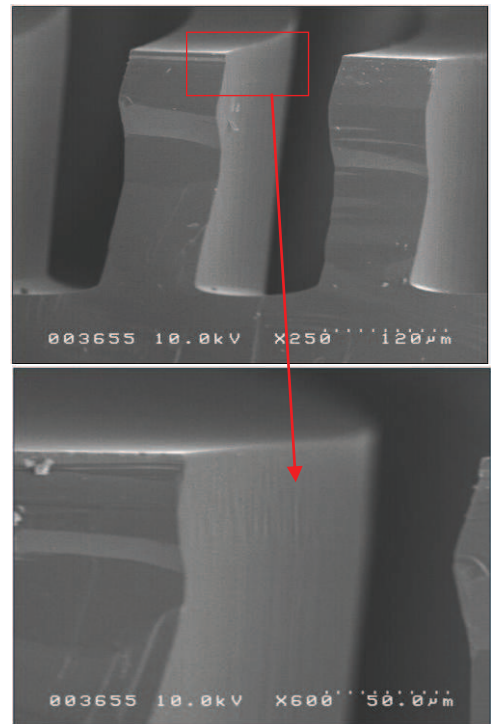


Fig. 5: SEM photograph of DRIE sidewall with annealing

## EVALUATION OF STORED ENERGY DENSITY

The maximum stored energy densities of the springs with/without the hydrogen annealing were evaluated by winding them using a wrench, as shown in Fig. 6.

Figure 7 plots the relationship between the winding angle and applied torque of Type B\_R. In the case of conventional metal coil springs, the sharp torque increments are generally observed around the winding limit. Also for the silicon spring with the hydrogen annealing, that was confirmed around the designed limit. Its maximum torque measured 400  $\mu\text{Nm}$ . On the other hand, the unannealed spring was fractured when the torque reached about 180  $\mu\text{Nm}$ , and the sharp increment was not observed. Thus, the maximum winding angle was successfully increased by the hydrogen annealing process.

The winding angle vs. torque relationship for Type\_A coil springs are summarized in Fig. 8. The red marks indicate the theoretical limits assuming silicon fracture toughness. The theoretical fracture stress [MPa] is written with them. All torques and winding angles were increased by the hydrogen annealing treatment. In particular, for Types A\_S and A\_F, the angle and torque limits of the annealed springs were closed to those of the theoretical calculations. For Type A\_R, the annealing effect seems to be relatively small. The cause of the annealing-effect dependency on the design has not been clarified.

Fig. 9 shows the measured stored energy density vs. the types of coils. There are three types of width and four types of total length. It shows the energy density was increased regardless of the width and the total length of the coil. Table 3 summarizes the averages of the measured energy density for the springs. The stored energy increased 3.5 times in average and 5 times at the maximum by the hydrogen annealing.

Therefore, it is demonstrated that the surface smoothing effect of the hydrogen annealing can improve the rotating angle and torque limit, which leads to improvement of the energy storage capacity.

Table 3 Averages of stored energy density [ $\mu\text{J}/\text{mm}^3$ ]

| Annealing | TypeA S | TypeA R | TypeA F | TypeB R |
|-----------|---------|---------|---------|---------|
| Without   | 116.3   | 119.2   | 45.2    | 76.2    |
| With      | 384.7   | 251.5   | 338.2   | 207.1   |

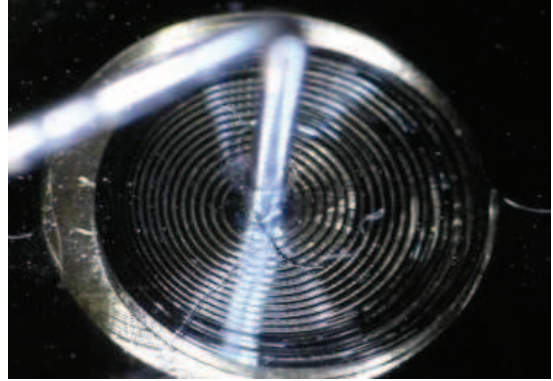


Fig. 6: Winding test of the silicon coil spring

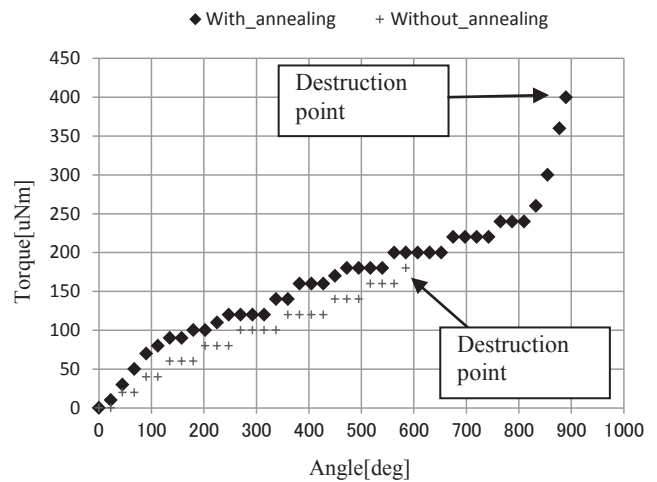


Fig. 7: Relationship between winding angle and torque of Type B\_R coil with and without annealing

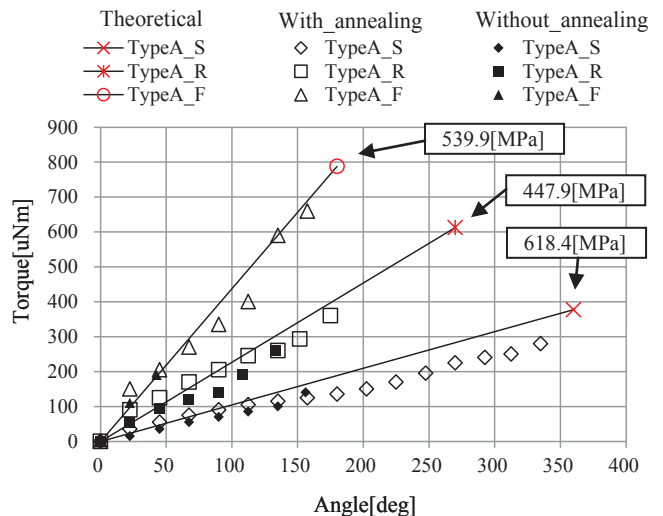


Fig. 8: Relationships between winding angle and torque of annealed and non-annealed Type\_A coils.

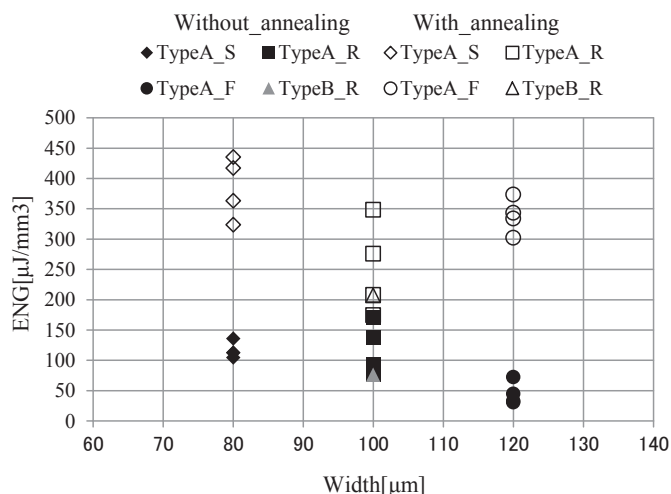


Fig. 9: Comparison of the stored energy densities of the annealed and unannealed springs.

### DESTRUCTION POINT OF COIL SPRINGS

In order to specify beginning of the destruction point, the stress distribution in fully winding was simulated. Figure 10 shows the simulation result of Type A\_R coil. The maximum stress is seen to be applied on the outer surface at the middle length from the center of the coil. However, the coil spring was destroyed at not only there but also other areas, as shown in Fig. 11. In the destruction, the shock at the middle was speculated to be propagated to the other area, which resulted in the multiple-points destruction.

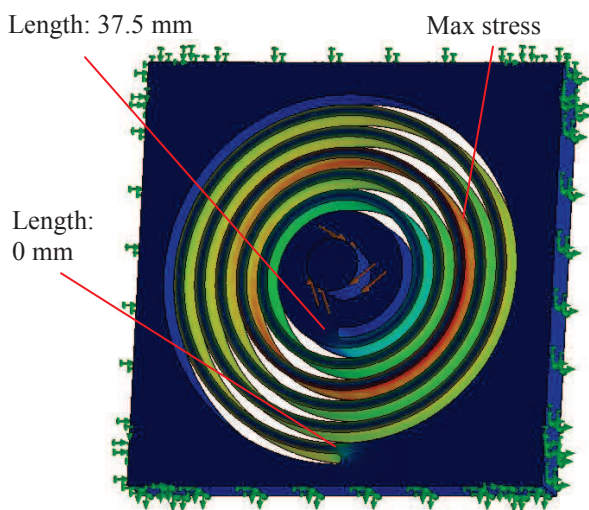


Fig. 10: Simulated stress distribution on Type A\_R coil spring. The red color indicates the maximum stress.

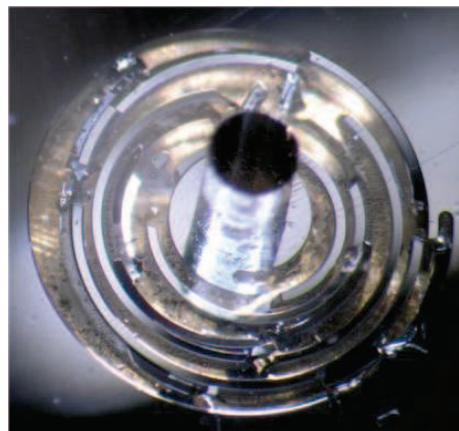


Fig. 11: Destroyed Type A\_R coil spring

### CONCLUSION

We demonstrated the hydrogen annealing process was effective to increase the fracture toughness of the silicon coil springs made by Bosch process. The stored mechanical energy could be increased about 3.5 times in average and 5 times at the maximum by the annealing. We believe that this process can provide a high-performance silicon coil spring enough to be applied to practical applications as an energy storage device.

### ACKNOWLEDGMENT

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### REFERENCES

- [1] “Non-Battery Energy Storage System using Coil Spring” NEDO exhibition in Innovation Japan 2011
- [2] “Realization of MEMS-based Si microscopic spring structures” Nanotech Japan 2012
- [3] Yoshiaki Matsushita et al.: “Effect of Hydrogen Anneal on Silicon Crystal” Vol.26 No.3 1999, The Japan Association for Crystal Growth
- [4] Kanamori et al.: “Development of a compact vacuum- and hydrogen-annealing machine for surface transformation of silicon and its applications to micro-optical devices” J. Vac. Sci. Technol. A, Vol. 26, No. 3, May/June 2008, American Vacuum Society