Supercritical carbon dioxide process for releasing stuck cantilever beams*

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Abstract: The multi-$SCCO_2$ (supercritical carbon dioxide) release and dry process based on our specialized $SCCO_2$ semiconductor process equipment is investigated and the releasing mechanism is discussed. The experiment results show that stuck cantilever beams were held up again under $SCCO_2$ high pressure treatment and the repeatability of this process is nearly 100%.

Key words: $SCCO_2$; sticking effect; release; cantilever beam; MEMS
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1. Introduction

Cantilever beams are widely used in MEMS structures and devices[1−3] because they are easy to fabricate. However, they are often attached to the substrate after wet etching of the sacrificial layer due to strong capillary forces[4]. Some researchers have investigated the mechanism of the sticking effect[5] and proposed the theoretical analysis of sticking phenomena. Mastrangelo[6] examined the deflection, mechanical stability and adhesion of thin micromechanical structure under capillary forces. Lin[7] discussed sticking phenomena on a cantilever beam with wetting length of liquid less than the beam length after spinning/drying. Lin and Chen[8] derived an adhesion criterion for center-anchored circular plates in microstructures. Besides theoretical research, several efficient sticking-free release processes were developed, such as XeF_2 dry release process[9] and $SCCO_2$ release process[10]. No matter the theoretical analysis and experimental sticking-free release processes, they both try to avoid the sticking effect during the microfabrication of cantilever beams. For cantilever beams which have already attached to the substrate, they are believed failed forever and cannot be processed in the next processing.

In this work, based on our $SCCO_2$ semiconductor process equipment, we explored the release processes for stuck cantilever beams. A multi-$SCCO_2$ release and dry process is proposed to completely release stuck cantilever beams. It turns out that this release process can be a master solution for all wet processes in MEMS/NEMS fabrications.

2. Experiments

In order to explore release processes based on $SCCO_2$, cantilever beams with the same width and different length were fabricated using MEMS microfabrication processes. The fabrication steps are shown in Fig. 1. Before the fabrication of cantilever beams, the RCA cleaning process was used to clean the wafer. First, 0.5 μm SiO_2 sacrificial layer was deposited by PECVD process. Then, it was patterned to define the anchor of the cantilever beam. After that, 0.5 μm SiN_x structural layer was deposited by PECVD process and patterned to define the cantilever beam. Finally, the SiO_2 sacrificial layer was released in BOE wet etchant.

The $SCCO_2$ release processes were conducted on our specialized $SCCO_2$ semiconductor process equipment shown in Fig. 2. Figure 3 shows the process chamber where wafers are held. The equipment contains the following main components. A refrigeration system keeps CO_2 in liquid phase and a heating system before the chamber allows high temperature and pressure CO_2 gas to be ventilated to the chamber. A CO_2 delivery system is among the entire equipment with a precise flow rate control unit which can be used to convey CO_2 in gas, liquid and supercritical state. A pump is added before the chamber which can compress CO_2 to a supercritical state. The process chamber is the place where the release and dry processes take place. It is capable of heating and cooling and the temperature and pressure can be controlled precisely. A backpressure regulator behind the chamber allows for rapid depressurization of the chamber manually. The whole equipment is controlled by a center computer.

The standard $SCCO_2$ release and dry processes based on our equipment are as follows:

(1) After releasing 0.5 μm SiO_2 sacrificial layer by BOE, the wafer was transferred to the chamber with an ethanol ambience.

(2) Liquid CO_2 was delivered to the chamber to replace ethanol. Through time control and observation from the quartz

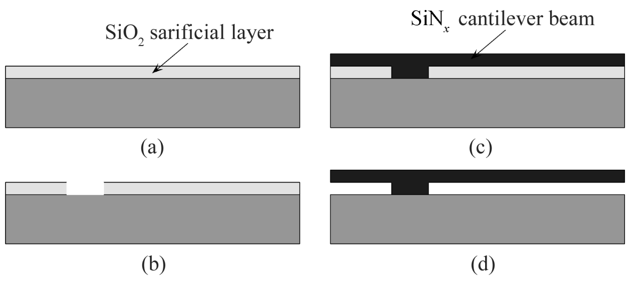

Fig. 1. Microfabrication steps for cantilever beams. (a) SiO_2 sacrificial layer preparation. (b) Anchor of cantilever beam definition. (c) SiN_x structural layer deposition. (d) Sacrificial layer releasing.

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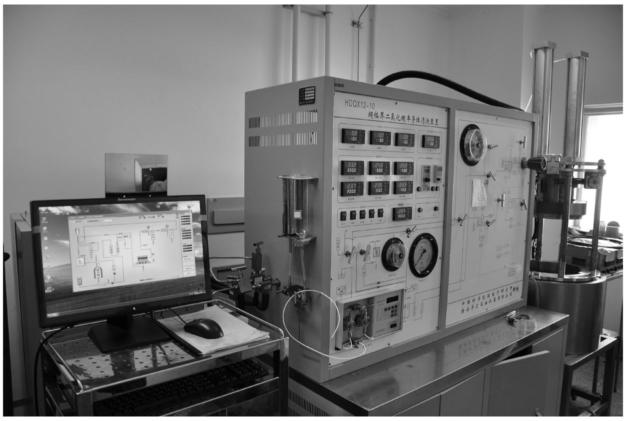

Fig. 2. $SCCO_2$ semiconductor process equipment.

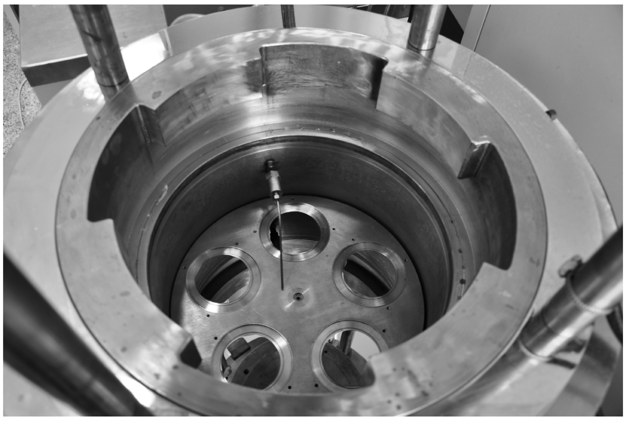

Fig. 3. Process chamber.

window on the chamber, when the chamber was full of liquid CO_2 without any ethanol, the delivery of CO_2 was stopped.

(3) By temperature and pressure control, CO_2 was kept beyond its supercritical point (31.2 ℃, 7.3 MPa).

(4) The chamber was depressurized gradually to 0.1 MPa at rate of 0.6 MPa/min and CO_2 was transformed from supercritical state to gas.

(5) The chamber was cooled to room temperature and the wafer was taken out.

The stuck cantilever beams were treated under multi-$SCCO_2$ release and dry process. Detailed steps of multi-$SCCO_2$ release and dry process are listed as follows:

(1) The wafer with stuck cantilever beams was cleaned by DI water then delivered to the chamber.

(2) Liquid CO_2 was conveyed to the chamber. When the wafer was completely immersed in liquid CO_2, the delivery of $CO_2$was stopped.

(3) The chamber was heated gradually from room temperature to 31.2 ℃ and the pressure of the chamber was kept beyond 7.3 MPa. Liquid CO_2 was transformed to supercritical state.

(4) The chamber continued to be heated and the temperature was kept above 50 ℃ and the pressure above 10 MPa. This state was maintained for 2 h.

(5) The chamber was depressurized rapidly from 10 to 0.1 MPa at rate of 0.6 MPa/s while keeping the temperature constant.

Table 1. Dimensions of the fabricated MEMS cantilever beams.

Parameter	Value
Cantilever width (μm)	10
Cantilever length (μm)	20–300
Cantilever thickness (μm)	0.5
Gap between cantilever and substrate (μm)	0.5

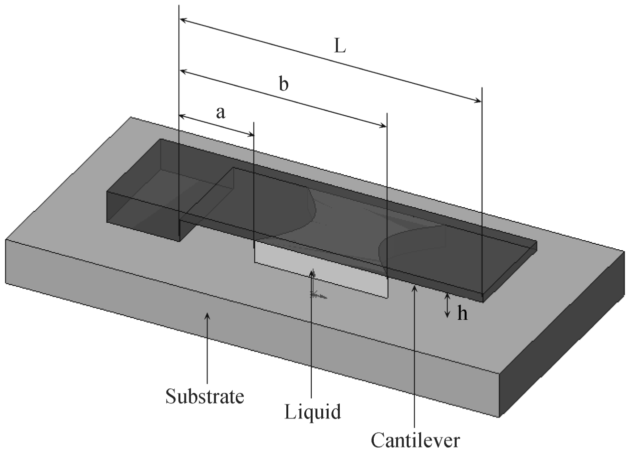

Fig. 4. Sticking model for cantilever beams.

(6) The chamber was cooled to room temperature and the wafer was taken out.

3. Results and discussion

In this work, various dimensions of cantilever beams were fabricated to test the different release processes. The parameters of the fabricated cantilever beams are given in Table 1.

The process which causes the sticking effect is the final release and dry process. According to the sticking model in Fig. 4 proposed by Lin[7], the adhesion criterion to determine whether the cantilever beam is sticking or not is

$$h_{\mathrm{c}} = \sqrt{\frac{\gamma \cos\theta_{\mathrm{c}}}{Et^3}(4b^3L - b^4 - 4La^3 + a^4)}, \qquad (1)$$

where γ is the surface tension between liquid and air, E is Young's modular, and θ_{c} is the contact angle. The liquid locates from a to b between the cantilever beam and the substrate. The length, width and thickness of the cantilever beam are L, w and t, respectively. In this experiment, the cantilever beam is SiN_x thin film and E is 300 GPa. The liquid is DI water and γ and $\cos\theta_{\mathrm{c}}$ are 7.275×10^{-2} N/m and 0.45, respectively. At the beginning of the dry process after releasing the sacrificial layer, suppose the DI water fills the entire length L between the cantilever beam and substrate. For the shortest fabricated cantilever beam ($L = 20$ μm), the calculated adhesion criterion h_{c} according to Eq. (1) is 0.647 μm. As the designed gap between the cantilever beams and substrate is 0.5 μm, all of the beams will be inevitable to attach to the substrate. Figure 5 shows the sticking effect under this situation.

If $SCCO_2$ release and dry process is used instead of the traditional wet etch and dry process mentioned above, the sticking effect can be avoided. The perfectly released cantilever beams are shown in Fig. 6. Regular interferometric fringes

Table 2. Physical properties of CO_2 in different phases.

Phase	Diffusivity (cm^2/s)	Viscosity ($mN{\cdot}s/m^2$)	Density (kg/m^3)
Liquid	10^{-5}	1	10^3
Supercritical fluid	10^{-3}	10^{-2}	10^2
Gas	10^{-1}	10^{-2}	1

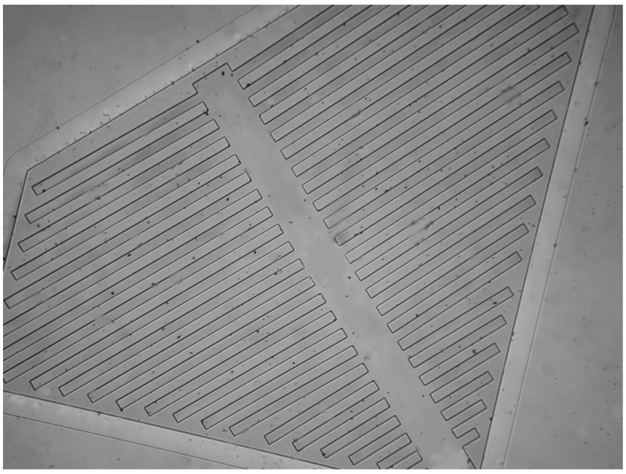

Fig. 5. Microscope photo of stuck cantilever beams.

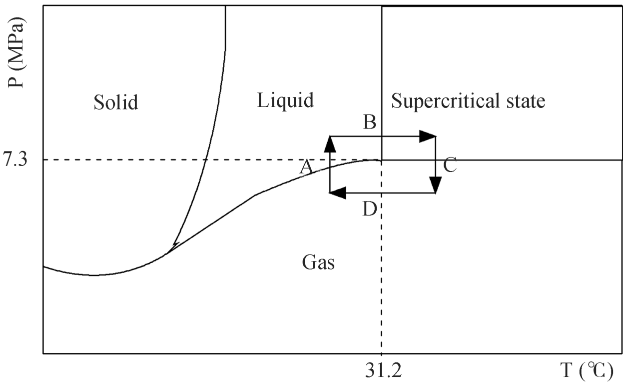

Fig. 7. States transformation of CO_2 during release and dry process.

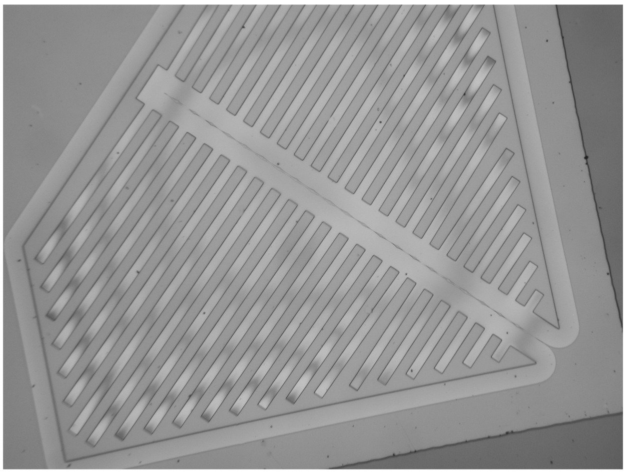

Fig. 6. Completely released cantilever beams using $SCCO_2$ release and dry process.

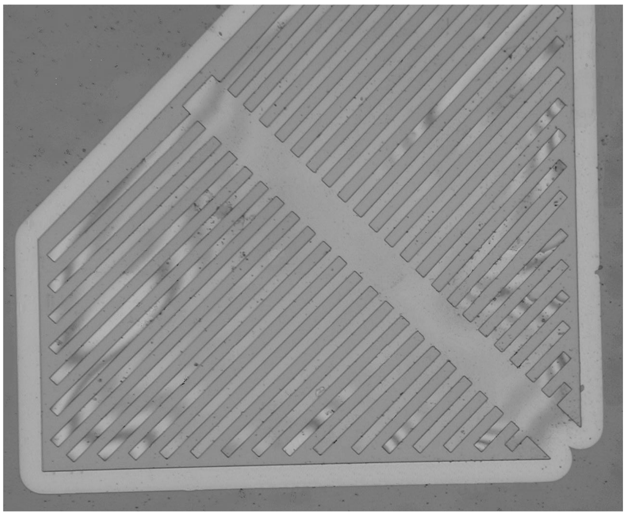

Fig. 8. Stuck cantilever beams were released again under multi-$SCCO_2$ release process.

indicate that the cantilever beams have been released completely without sticking. The main reason that the sticking effect can be avoided is that the surface tension of $SCCO_2$ stays at zero beyond the critical point and liquid droplets are no longer formed[11]. The phase transformation of CO_2 during the release and dry process based on our equipment is illustrated in Fig. 7. 99.9999% CO_2 gas from the cylinder was refrigerated to liquid along curve A before it was delivered to the chamber to replace the solvent. When the chamber was full of liquid CO_2, it was heated and CO_2 reached supercritical state along curve B. When the drying process began, the chambers pressure was reduced while keeping the temperature constant along curve C. Before the wafer was taken out, the chamber was cooled to room temperature along curve D. During the whole process, no transformation of any solvent from liquid to gas was generated, so no interface of liquid-gas was formed and the sticking effect was completely avoided.

The results of multi-$SCCO_2$ release and dry process for stuck cantilever beams are shown in Fig. 8. Interferometric fringes appeared again, which indicate that once attached cantilever beams have been held up. So the stuck cantilever beams were released again under the treatment of multi-$SCCO_2$ release and dry process. 36 groups of cantilever beams (44 cantilever beams make up one group) were tested and the results showed that only several of them failed to release. $SCCO_2$ has diffusion properties similar to gas and solution properties similar to liquid. Table 2 compares the physical properties of CO_2 in different phases. The perfect transportation property of $SCCO_2$ allows CO_2 molecules to penetrate and reach any space whose volume is bigger than that of CO_2 molecules under high pressure[12]. The mechanism of releasing stuck cantilever beams by

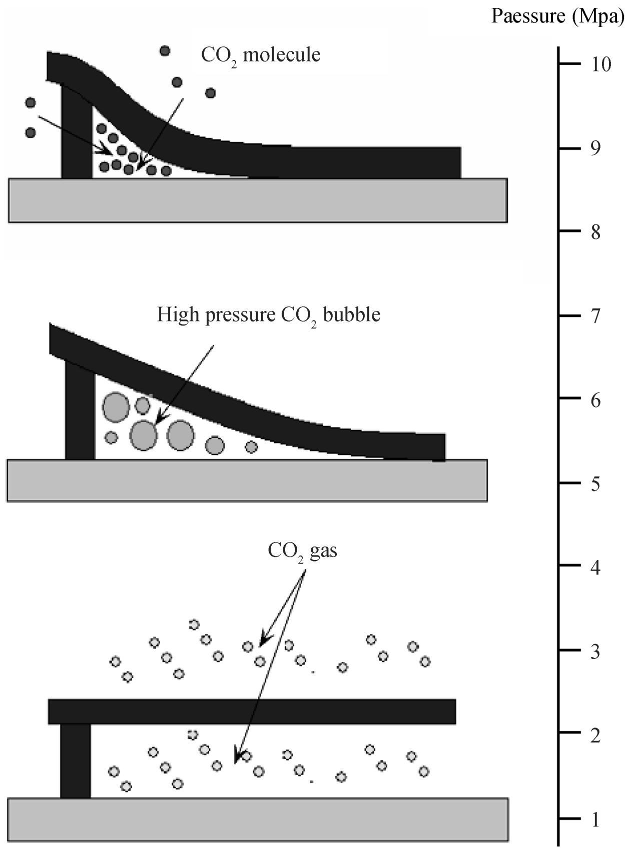

Fig. 9. Mechanism of $SCCO_2$ releasing stuck cantilever beams.

high pressure $SCCO_2$ is illustrated in Fig. 9. $SCCO_2$ molecules move to the narrow space at the anchor of the cantilever under high pressure. As long as the pressure is high and $SCCO_2$ is sufficient, the space of the anchor can be filled with high density of $SCCO_2$. Then, the chamber is rapidly depressurized, CO_2 transforms from supercritical state to gas. During this process, high pressure CO_2 bubbles (6–7 MPa) are formed and stuck cantilever beams are held up under the force resulting from the formation of CO_2 bubbles. The entire process is very similar to opening a bottle of soda water, when a great deal of CO_2 bubbles generate and large force is caused. Once the cantilever beams are released, they will not stick to the substrate because the chamber is now full of CO_2 gas and no interface of liquid-gas is formed. This process is repeatable, which means if the cantilever beams attach to the substrate again under capillary force, they can be held up again under high pressure $SCCO_2$ treatment. This is because there is always a cavity at the anchor of the cantilever which can be filled by $SCCO_2$ molecule. Generally speaking, any microstructure with a cavity which forms during the release and dry process under capillary force can be released again under the multi-$SCCO_2$ release and dry process.

4. Conclusion

In this paper, $SCCO_2$ release processes are investigated and a multi-$SCCO_2$ release and dry process is proposed to release stuck cantilever beams. The $SCCO_2$ release process is explored based on our $SCCO_2$ semiconductor process equipment. Experiment results show that once stuck cantilever beams were released completely under the multi-$SCCO_2$ release process treatment. This novel releasing process can be used to release failed cantilever beams and other microstructures caused by the sticking effect. It is an efficient method to rescue stuck microstructures in MEMS fabrication.

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