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Supercritical drying process for high aspect-ratio HSQ nano-structures

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Abstract

Supercritical resist drying allows the fabrication of high aspect-ratio (AR) resist patterns. The potential of this drying technique to increase the maximum achievable AR and the resolution of the overall lithographic process is analyzed for hydrogen silsesquioxane (HSQ). The maximum achievable AR is doubled compared to conventional nitrogen blow drying. Furthermore, the resolution is improved significantly.

Keywords: Supercritical resist drying; HSQ; High-resolution electron beam lithography; Aspect ratio

1. Introduction

Collapse of high aspect-ratio (AR) resist patterns is a serious problem associated with the fabrication of nano-structures. Pattern collapse often occurs after the development process step during resist drying, because the surface tension of the liquid remaining between two adjacent structures pulls the lines together [1]. A very effective method to optimize resist drying is the replacement of the rinsing liquid by a supercritical fluid to reduce the surface tension [1,2]. The aim of this work is to compare supercritical resist drying (SCD) techniques using carbon dioxide (CO_2) with the conventional resist drying by nitrogen (N_2) blow and explore the potentials for the fabrication of nano-structures with high ARs. The reasons for using CO_2 as supercritical fluid is that the critical point is reached at a moderate temperature and pressure of $T_c = 31.1$ °C and $P_c = 7.38$ MPa, respectively [2].

2. Experimental

High AR resist structures have been fabricated in hydrogen silsesquioxane (HSQ) as high-resolution negative tone electron beam resist [3]. Silicon samples have been spin coated with HSQ to thicknesses of 190 nm and 770 nm. Patterns consisting of pairs of lines as well as of isolated lines have been exposed with a Leica EBPG-5000 electron beam system at 100 keV and various doses. Immediately after exposure, the development has been carried out at 21 °C with high concentration tetra-methyl ammonium hydroxide (TMAH) [4]. After development all samples have been rinsed in DI water and subsequently in 2-propanol. Sample drying has been carried out using a SCD technique and, for comparison, by conventional nitrogen blow.

For SCD a supercritical resist dryer constructed by *SC Fluids* [5] has been used. The wet HSQ samples are placed in the process chamber of the dryer, which is filled up with 2-propanol to avoid any resist drying before reaching the supercritical state. The pressure is then increased by introducing liquid CO_2 at a pressure of 7 MPa while keeping the temperature at 21 °C. Next, the temperature is raised to 35 °C leading to higher chamber pressure. During this procedure, the condition becomes supercritical. In a last step, the supercritical CO_2 is slowly released and pressure and temperature are lowered to ambient conditions.

Observations of the developed resist patterns have been carried out with a high-resolution Leo DSM 982 Gemini

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scanning electron microscope (SEM). In order to determine the aspect ratios from the heights and the widths of the lines, samples have been cleaved orthogonal to resist structures and their cross-sections thoroughly studied.

3. Results and discussion

This section of the paper is divided into two main parts. In the first part, the maximal achievable ARs for pairs of HSQ lines with different width and a fixed height of $H = 190$ nm are determined. In the second part, the resist thickness is increased to $H = 770$ nm. Again the maximal achievable ARs have been obtained. Additionally, various line spacings and isolated lines have been investigated. For both HSQ layer thicknesses the results obtained with the SCD process are compared with those for conventional nitrogen blow.

First, pairs of lines with a height of $H = 190$ nm and a fixed line spacing of $d = 100$ nm have been investigated. Using nitrogen blow, a maximum AR of 8 has been achieved. An example of such a pattern with line widths of $W = 23$ nm is shown in the SEM image in Fig. 1.

Patterns collapse for line widths below 23 nm, if N_2 blow is used as drying technique. In Fig. 2, collapsed lines with $W = 14$ nm are shown as an example.

Identical 14 nm lines dried with the SCD process are shown in Fig. 3. Here, no pattern collapse has been observed at all and an AR of about 14 has been achieved. The comparison of Figs. 2 and 3 clearly demonstrates the decisive influence of the SCD process on the maximum AR.

In order to determine the maximum achievable ARs for both drying techniques, a systematic variation of the line width between 10 and 30 nm has been performed. For better statistics, several samples have been analyzed. From these investigations, the probability of pattern collapse at a certain line width has been determined.

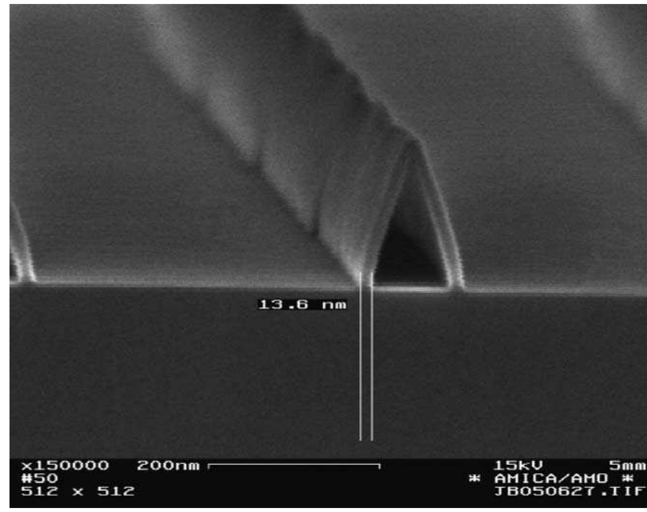

Fig. 2. SEM image of collapsed HSQ lines after conventional drying.

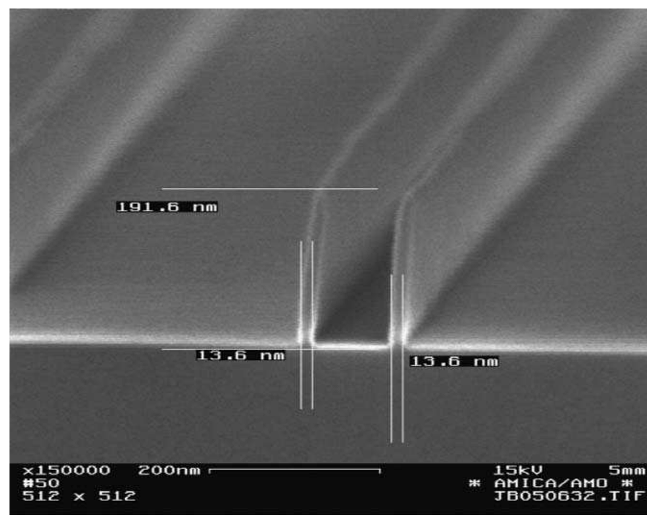

Fig. 3. Pair of 14 nm HSQ lines with an AR of 14, dried by SCD.

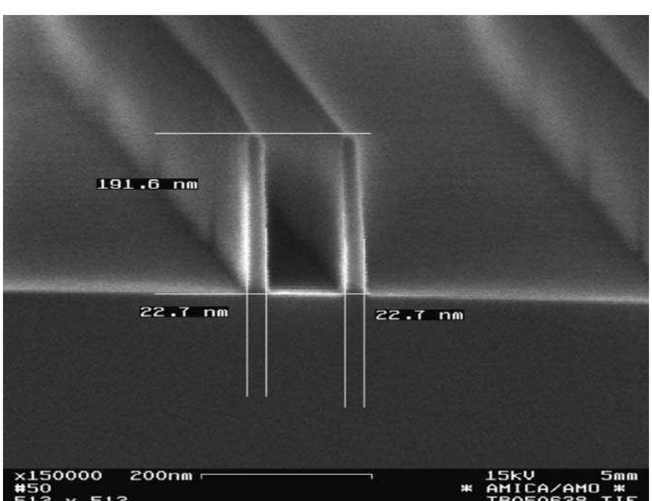

Fig. 1. Pair of 23 nm HSQ lines with an AR of 8 after conventional drying.

The results are summarized in Fig. 4, where the probability of pattern collapse is shown as a function of the AR for pairs of lines with a spacing of 100 nm. For conventional processing with N_2 blow, represented by the solid squares in Fig. 4, the ARs where no pattern collapse is observed are below 7.5. Increasing the AR leads to a strong increase of pattern collapse probability. At an AR of 9 already 90% of the lines are collapsed. Compared to the nitrogen blow, the maximum AR is doubled using the SCD technique, as the hollow circles in Fig. 4 show. Again, a sharp transition between the regimes of no line collapse and 100% collapse is observed.

With an etch selectivity of about 3 between silicon and HSQ in HBr chemistry based RIE etching processes, the HSQ thickness of 190 nm is sufficient to act as etch mask for transfer of well-defined patterns to a substrate and

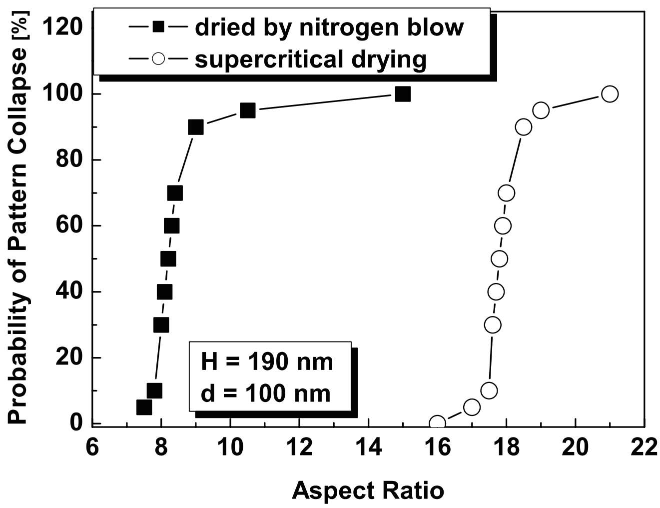

Fig. 4. Probability of pattern collapse vs. AR.

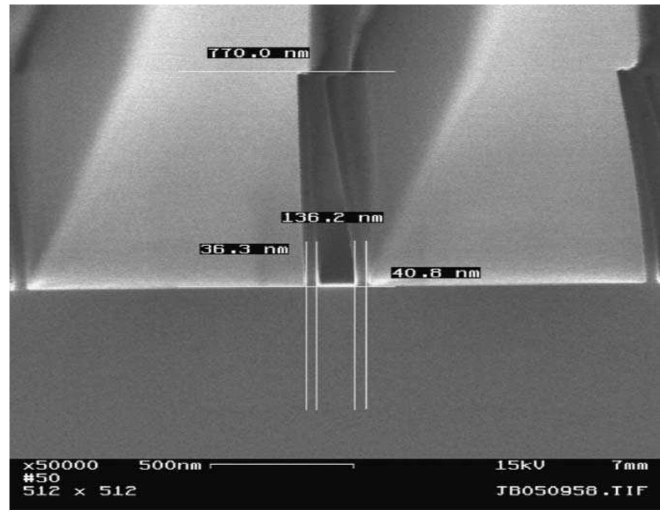

Fig. 6. Pair of 40 nm HSQ lines with an AR of 20, dried by SCD.

realize functional devices for a wide range of applications [6]. These results clearly demonstrate the benefits of SCD to fabricate structures in the 10 nm regime with this resist thickness.

To explore the limits of SCD processing, the HSQ thickness has been increased to 770 nm. Compared to the investigations presented above, in this case not only the line width but also the line spacing has been varied.

Using nitrogen blow as a drying technique, the minimum spacing between the lines of the pair is 170 nm. At a line width of 68 nm an aspect ratio of 11 has been achieved, as shown in Fig. 5. For line spacing lower than 170 nm the lines either collapse or are not completely resolved. Similarly, reducing the line width leads to pattern collapse. Using the SCD process, the minimum spacing between pairs of lines has been reduced down to 135 nm and the maximum achievable aspect ratio increased to 20, as shown in Fig. 6. These results imply that not only the AR, but also the resolution of the overall lithographic process can significantly be improved by SCD.

In densely packed resist structures pattern collapse is commonly attributed to the surface tension of the rinsing liquid remaining between the structures [1]. This pattern collapse mechanism, however, cannot be operative for isolated lines. Nevertheless, the process of supercritical resist drying is beneficial also for isolated lines [7].

In the present experiments, the already impressively high AR of 35 obtained for isolated lines with the conventional drying technique has further been increased to AR = 44 with the SCD process. A line with an extremely small line width of $W = 18$ nm at the height of 770 nm is shown in Fig. 7. The improved AR of isolated lines after supercritical drying process could be attributed to the extremely high diffusivity of the supercritical CO_2 [7].

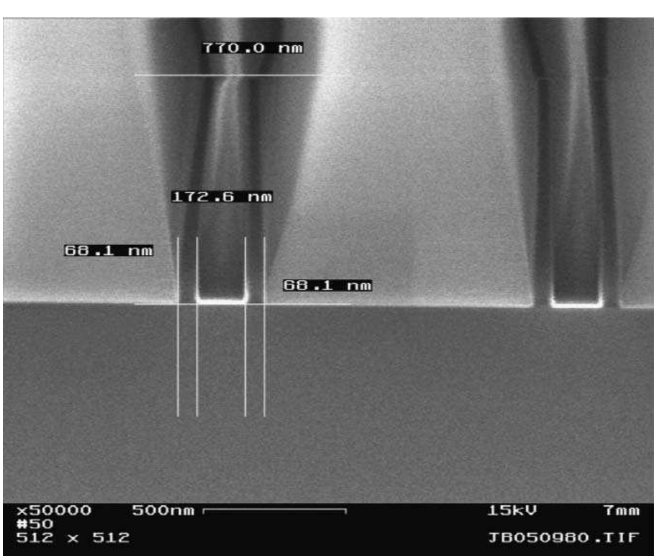

Fig. 5. Pair of 68 nm HSQ lines with an AR of 11, dried by N_2 blow.

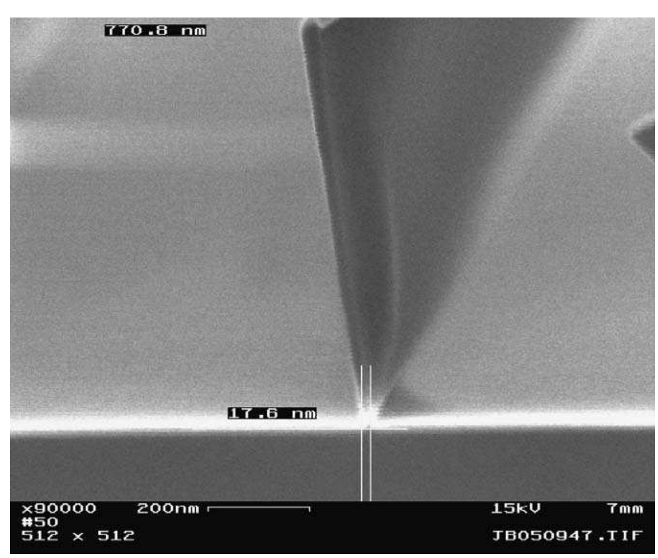

Fig. 7. Isolated HSQ line with AR = 44, dried by SCD.

Therefore, supercritical CO_2 can easily penetrate into the resist polymer matrix replacing the existing rinsing liquid and during resist drying flow out of lithographic structures without causing any tension within the material. As a result, lower roughness and improved stability of the structures is achieved [7].

4. Conclusion

A SCD process for HSQ using CO_2 as supercritical fluid has been presented. The maximum AR for 190 nm high SCD dried resist is twice as high as for N_2 blow dried resist. For a higher resist thickness of 770 nm a similar increase of the AR by a factor of two has been achieved. Even for isolated lines, where surface tension is ruled out as a source for pattern collapse, SCD has a beneficial effect and increases the maximum AR considerably. Furthermore, SCD processing shows great potential to improve the resolution in dense nano-patterns.

Acknowledgment

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