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Preparation of Silica Fibers from Sodium Silicate Solution

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Silica fibers were prepared from sodium silicate solution. A spinnable sol was obtained by the reaction of a sodium silicate solution with ethyl acetate, and gel fibers obtained by dry spinning of this sol were converted to silica fibers by treating in an acid bath followed by drying at 100 °C and heating at 600 °C.

In recent years, a sol-gel process for preparing silica glass fibers using a silicon alkoxide as a raw material has been focused.[1] A main advantage of this process is the reduction of energy consumption, since this process could be operated at a much lower temperature than that employed in a conventional quartz-fusion process. However, such alkoxide-derived fibers are still rather expensive. Recently, Achtsnit[2] has developed a process for the production of silica fibers from an aqueous sodium silicate solution, so-called water glass(abbreviated as WG). Sodium silicate gel fibers were drawn by dry spinning of a high-viscosity sol obtained from a WG, and then were converted to silica glass fibers by an acid-treatment and a heat-treatment. This process is attractive from the viewpoint of cost saving by using a WG in stead of expensive alkoxides. But unfortunately, further details of his method, especially of preparing such a highly viscous sol have not been reported.

We have found that the addition of a viscosity-increasing reagent such as ethyl acetate to a WG resulted in a spinnable sol, which could successfully be converted to highly pure silica glass fibers by treating the sol in almost similar manner as above. Even if this method is not an alternative, it is still important, because our findings will give a clue to the elucidation of Achtsnit's method.

A mixture of WG(No.2) and a viscosity-increasing reagent with a different volume ratio was vigorously stirred at a room temperature or around 40 °C for 3-4 h in an open system. When the sol thus obtained became sticky and spinnable, gel fibers were drawn by dipping a glass rod into the sol and pulling it up manually, and then immersed in an aqueous solution of nitric acid, followed by drying at 100 °C for 15 min and heating at a temperature above 600 °C for 1 h to give silica fibers.

It was found that ethyl acetate was best as a viscosity-increasing reagent. The duration for possible fiber drawing was very short when methyl acetate was

employed, and gelling took place immediately in the case of ethylene chlorohydrine. Therefore, ethyl acetate was used in this study. The composition of the starting mixture was a key factor in order for the fiber spinning to be possible for long time. Unless otherwise stated, the best composition, 1:1 volume ratio of the WG to a viscosity-increasing reagent was adopted. The time required for the starting mixture using ethyl acetate to reach a spinnable state at a room temperature was about 3 h but it became shorter with increasing reaction temperature.

The acid-treatment of gel fibers was necessary to produce water-insoluble gel fibers and to obtain heat-stable silica glass fibers. Without the acid-treatment silica fibers melted at 700 °C. Figure 1 shows a scanning electron micrograph of a typical silica fiber. As clearly seen, it had smooth surface, and the shape of cross section was almost circular. The silica fibers of ca. 60 cm long and ca. 10 μm diameter were readily obtained. It was confirmed by X-ray diffraction profiles that the silica fibers were generally amorphous up to 1000 °C. However, a small amount of cristobalite precipitated in the resultant silica glass fibers when the time of the acid-treatment was shorter, and/or the concentration of the acid solution was lower. In addition, the IR spectra of the silica fibers heated at 800 °C were quite identical to that of a commercial quartz wool. Table 1 lists selected results of the electron probe microanalyses of various silica fibers. It is surprising that the amount of remaining sodium in silica fibers prepared by using ethyl acetate was less than identification limit (estimated at 0.01 wt%). On the other hand, the silica fibers prepared in the absence of ethyl acetate contained a trace amount of sodium in spite of a thorough acid-treatment (6 M, 18 h). Such a different behavior in sodium removal will be ascribed to the essential difference between the states of the two kinds of spinnable sols; ethyl acetate is considered to play a certain role for the occurrence of the easy removal of sodium. Details of the role of ethyl acetate will be published separately.

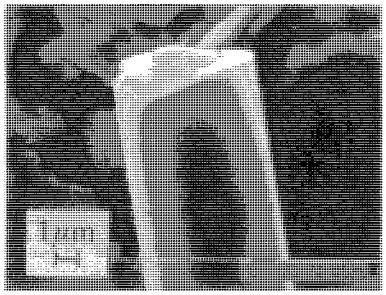

Fig. 1. Scanning electron micrograph of a silica fiber prepared by treating in 0.1 M HNO_3 for 1min and by heating at 1000 °C for 1 h.

Table 1. EPMA analyses of silica fibers heated at 1000 °C[a)]

No.	Ethyl acetate	Acid-treatment (conc./M, time/min)	Intensity(cpm) Si	Na
1	yes	6, 1080	41154.2	0.0
2	yes	6, 1080	40127.6	0.0
3	no	6, 1080	46991.8	2.5
4	no	6, 1080	46469.9	2.8
5	yes	0.1, 1	14600	0
6	yes	0.1, 1	15000	0
7	no	0.1, 1	14400	26
8	no	0.1, 1	14900	34

a) Measurements of No.5-8 were performed by using another instrument.

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References

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