FABRICATION OF PSZ FERRULES FOR OPTICAL CONNECTORS BY THE INJECTION MOLDING PROCESS

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ABSTRACT

In the fabrication of PSZ ferrules for optical connectors by the injection molding process, the molding die was optimized. Furthermore, the fabrication conditions of molding were evaluated in detail, and the material characteristics of the sintered sample were evaluated. By using a molding die with an air vent and two gate balance runners, production of highly reliable PSZ ferrules has been accomplished. From the viewpoint of mold shrinkage ratio, filling ratio and percentage of incomplete ejection, the optimal conditions for fabrication of injection molding samples are: a holding pressure of higher than 60MPa and less than 80MPa, a duration of dwelling of longer than 3 sec and less than 5 sec, and a compound viscosity of 1,000Pa.s. The PSZ ferrules for SMF fabricated by the injection molding process approximate the dimensional accuracy requirement and demonstrate a fairly high level of the near-net shape characteristics.

INTRODUCTION

The range of application of optical transmission systems using single-mode fibers (SMFs), which enable wide-area information transmission, is expected to expand to individual subscriber's systems; thus, economical production of the SMF optical circuit parts that are required for the expansion is an important issue. The optical connectors needed for connection between SMFs require the minimum optical loss characteristics without adjustment after connection. Therefore, they are fabricated very accurately, and the connection performance has almost reached the limits of possible improvement. The cost of ferrules in a connector set is high, more than 60 percent of the total cost; thus, from the standpoint of making SMF components more economical, it is important to reduce the cost of ferrules(1).

Alumina has traditionally been used for ferrules. Recently, however, partially stabilized zirconica (PSZ) has begun to be used because PSZ has higher flexural strength and fracture toughness; it also has a smaller Young's modulus and thermal expansion coefficients as well as good grinding and polishing characteristics. Thus, the production process can be simplified when PSZ is used(2). However, generally speaking, ferrules are produced by the extrusion molding process, and after sintering, a complicated grinding and polishing process must be performed for guiding optical fibers to tapered holes. Furthermore, PSZ is brittle compared with metals. Another disadvantage is that, with the use of PSZ, problems such as the development of grinding chips still remain after polishing.

The authors previously studied the packing conditions of alumina particles of ceramic powder and the characteristics of the sintered body obtained(3). At the same time, they reported on the fabrication of artificial polycrystalline ruby by the same method(4). In the present study, the modeling of molding dies and the near-net shapes which were not studied in the past were studied with respect to PSZ ferrule fabrication. The optimal relationships between the holding pressure during injection molding, duration of dwelling, and viscosity of ceramic compounds; and the shrinkage of molded samples and ejection characteristics, have been established. At the same time, a simple method to obtain the relationship between the holding pressure and mold shrinkage ratio in terms of Archimedes's method from the weight of the molded sample has been established. Also, the material characteristics of ferrules were evaluated. The results indicate that the ferrules produced by the process developed in the present study are reliable and economical. Thus, they can be used in practical applications.

EXPERIMENTAL METHOD

Partially stabilized zirconia (Tosoh, TZ-3Y) was used as a ceramic powder. Also, thermoplastic resin (Daiichi Kogyo Seiyaku), paraffin wax

(Nippon Seiro), and di-n-butyl phthalate (Wako Junyaku) were used as organic binder.

Figure 1 shows the flow chart of PSZ ferrule production. Thermoplastic resin (14.5wt%), paraffin wax (3.0wt%) and di-n-butyl phthalate (0.5wt%) (when the weight of ceramic powder was taken as 100wt%) were added to ceramic powder with benzene and mixed with a nylon ball mill. After 24hrs distillation drying at 373K, the mixture was heated and kneaded to uniformity by a ceramic roll mill (Noritake, NR-84D) and was made into a compound. The viscosity of the compound was controlled to the desired level by controlling the temperature during dwelling of the injection molding machine (Nippon Seikosho, JSW-15T). Also, the relationship between the holding pressure and mold shrinkage ratio in terms of Archimedes's method was examined. The molded sample was debound completely by heating to 723K and 0.8MPa of N_2 ambient for 72hrs using a pressurized debinding furnace (Tokai Konetsu, TJ-400 III). After debinding, the sample was calcined and treated with acid to remove metal contamination.[5] Then, it was sintered for 12hrs with a maximum

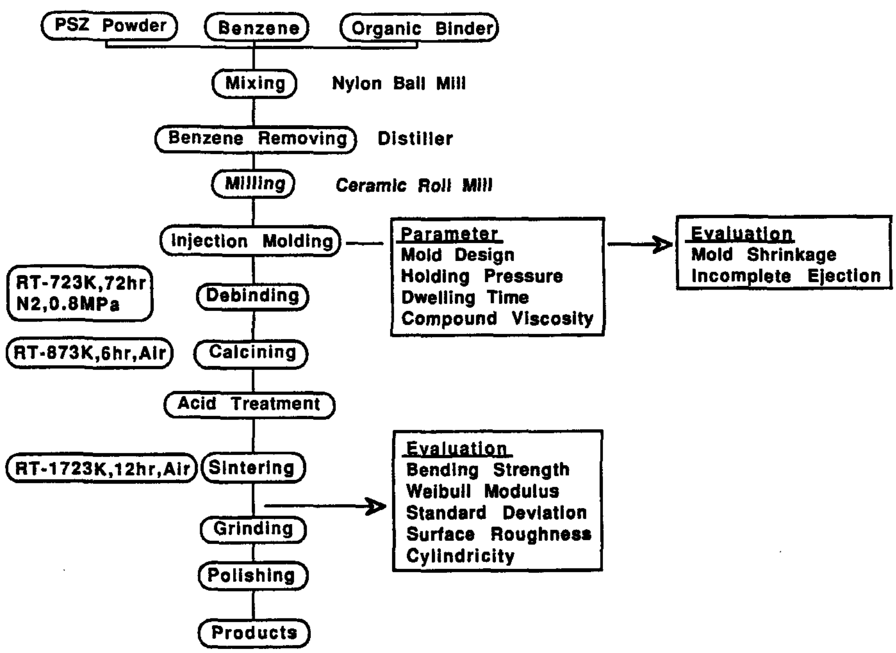


Figure 1. Flow chart of PSZ ferrule production.

temperature of 1723K under atmospheric pressure. After sintering, the molding die was evaluated in terms of flexural strength and Weibull modulus[6] by means of a three-point flexural testing machine (Shimazu, OSS 5000). Here, the samples tested were ferrules of 2.593mm external diameter and 0.124mm internal diameter. Measurements were carried out with a span of 10mm and at the cross head speed of 0.008mm/sec. Then, the dimensional characteristics were evaluated in terms of Standard Deviation (SD) due to shrinkage, surface roughness, and cylindricity. A scanning electron microscope (SEM) (Hitachi, S-800) was used to observe the sample conditions. An electronic balance (Mettler, TM4000) was used for weight measurements, and a microscope (Topcon, TMM-100D) was used for the measurement of dimensions. In addition, a cylindricity measuring instrument (Taylor Hobson, TALYROND 200) and a surface roughness gauge (Tokyo Seimitsu, SURFCOM 550A) were utilized.

EXPERIMENTAL RESULTS AND DISCUSSION

Molding Die

Schematic diagrams of the molding die when it is opened and closed and the molded samples of PSZ ferrules in the present study are shown in Fig. 2. The die features two symmetrical runners (a) and an air vent[7]. With the conventional single-runner die (b), deviation of pressure distribution develops on the sample; thus, it is difficult to maintain the dwelling function[8]. To avoid such uneven pressure distribution, as shown in the figure, two gate balance runners, symmetrical with respect to the cavity, were installed, and uniform compound flow and pressure distribution were established[9]. Also, generally speaking, air inclusion during injection molding leaves behind pores after sintering. These pores deteriorate the strength of the ferrules[10]. Therefore, in the present study, an air vent (shown in Fig. 2) is installed to remove air in the molded sample. Figure 3 shows a comparison of the fracture surface conditions observed by SEM, the Weibull modulus and the flexural strength, for the ferrule produced by the die with an air vent and the other ferrule produced without an air vent. As shown in the figure, the pores become the origin of fracture in the sintered sample which was produced by a die without an air vent;

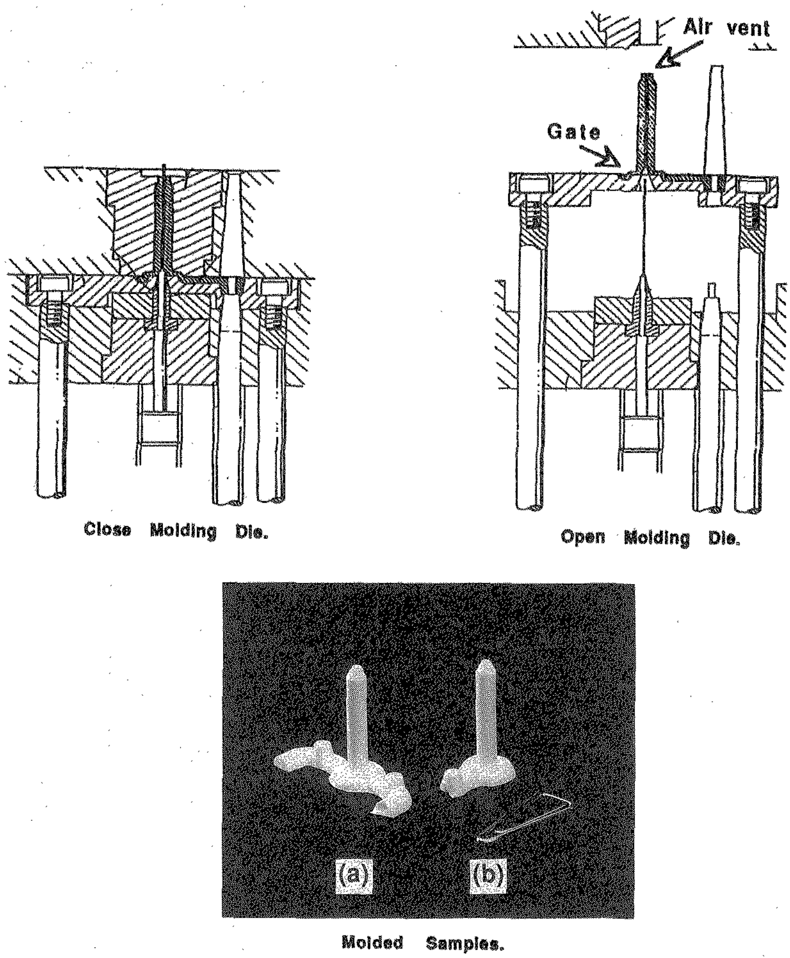


Figure 2. Schematic diagrams of the molding die when it is opened and closed and the molded samples of PSZ ferrules.
(a) obtained with two gate balance runners
(b) obtained with the conventional single runner

conversely, when the sample was produced by a die equipped with an air vent, the fracture originates at the surface of the sintered sample. This indicates that air in voids accumulates in the air vent and voids in the sample are eliminated by the air vent. Since the air vent was removed after sintering, the Weibull modulus and flexural strength of the ferrule produced without an air vent were small, 5.0 and 780MPa, respectively. However, the Weibull modulus and flexural strength of ferrules produced with an air vent were improved to 9.2 and 950MPa, respectively.

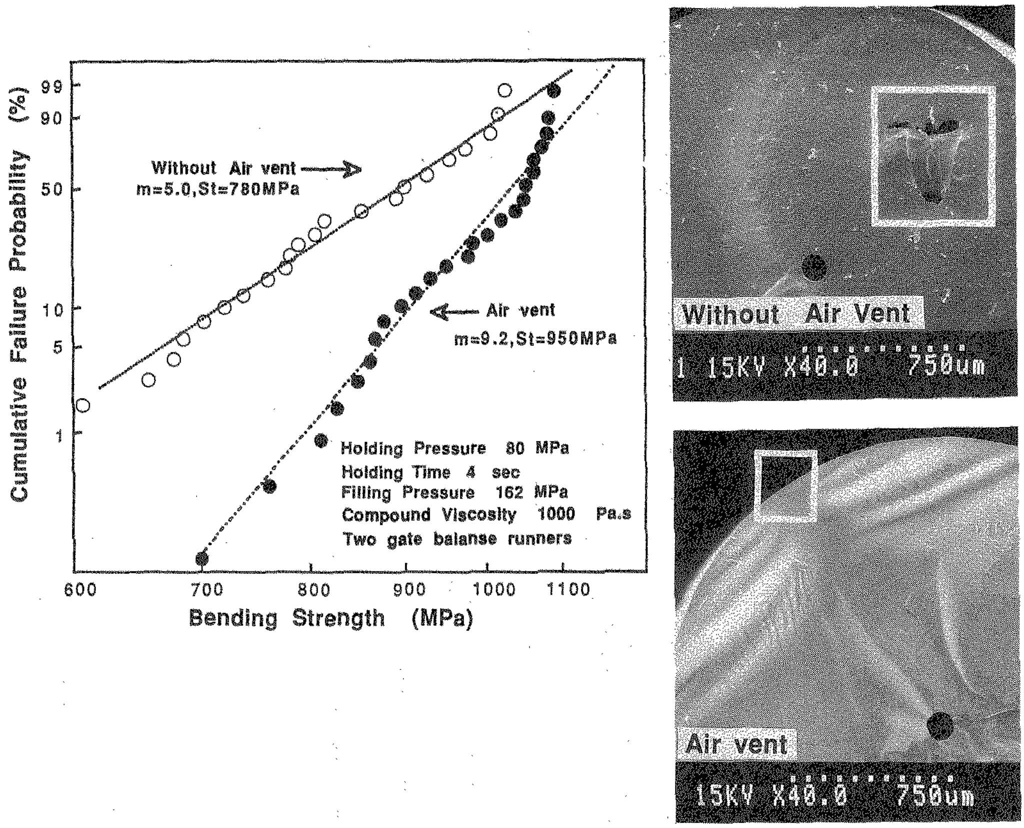


Figure 3. SEM photographs of the fractured surface and graph of the Weibull modulus and flexural strength for the ferrules produced by the die with an air vent and without an air vent.

In the experiment below, the production conditions of ferrules and the sample dimensions after sintering were evaluated using the samples produced by a die equipped with two symmetrical runners and an air vent.

Injection Molding Conditions Dwelling

Dwelling: Figure 4 shows the relationship between the sample dimensions, ϕ and L, and die dimensions, ϕ mold and L mold, and the holding pressure (hereinafter denoted as P). Here, the molding shrinkage ratios are defined as $S_1 = (\phi \text{ mold} - \phi)/\phi \text{ mold}$, $S_2 = (L \text{ mold} - L)/L \text{ mold}$. Furthermore, to evaluate the exclusive relationship between P and the molding shrinkage ratio, the viscosity of the compound and the dwelling time were kept constant. As shown in the figure, S_1 and S_2

decreased with the increase of holding pressure P. They are in a state of equilibrium in the region where P is above 60MPa, which may be due to the elastic properties and the volume reduction accompanied by solidification during cooling[11]. These results imply that if the equilibrium values of S_1 and S_2 are obtained in the binder type, the approximate dimensions of a die to produce a desired sample can be predicted. Also, the same figure shows the relationships vis-à-vis the holding pressure P of the molding shrinkage ratio in the radial direction, S_1, and that in the axial direction, S_2. With the increase in P, both S_1 and S_2 decrease; the rate of reduction of S_2 is larger than that of S_1. These values take identical values in the region above 60MPa, and are in equilibrium. This can be explained as follows: in the region where the holding pressure is above 60MPa, the repulsive force from the compounds pressed by the die acts on the succeeding compound, and the directionality of pressure distribution in the sample disappears. As a result, the orientation of the compound which was parallel to the incoming direction disappears and a nonoriented state develops[12]. In the figure, the effect of the number of runners on the holding pressure is added. As shown in the figure, the case of a

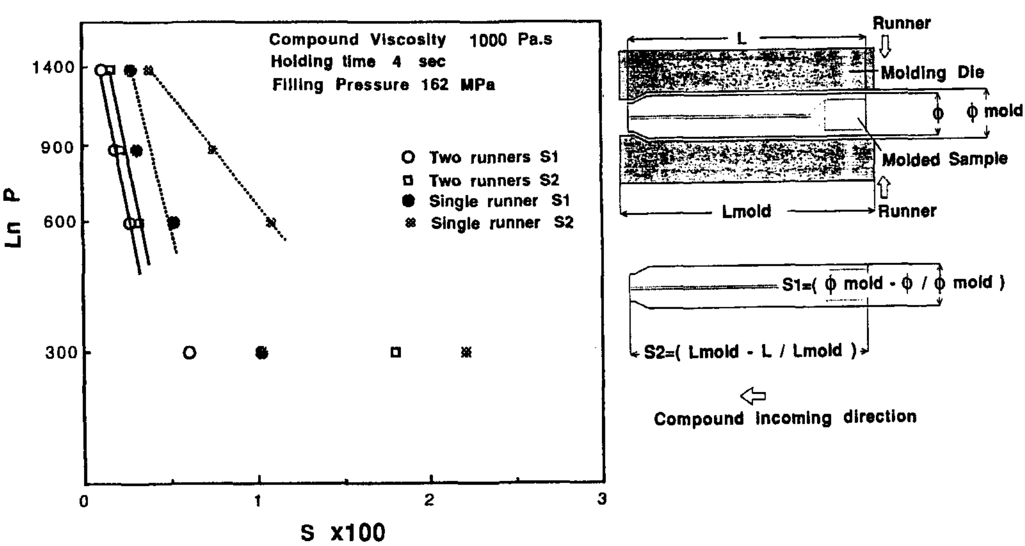


Figure 4. Relationship between the sample dimensions, ϕ and L, and die dimensions, ϕ mold and L mold, and the holding pressure.

single runner required higher holding pressure to obtain the same mold shrinkage ratio as in the case with two runners. Furthermore, the dwelling function of the single-runner die is inferior to that of the die with two runners.

Figure 5 shows the relationship between the holding pressure P and the filling ratio, $A=(\phi_a/\phi_b)$. As shown in the figure, the filling ratio A increases with the increase in P and reaches the equilibrium above 60MPa. Since the filling of the die with the compound advances from the runner side, when the holding pressure is low, shrink marks develop from the location away from the runner. With the increase in the holding pressure P, the number of shrink marks decreased. Thus, if a pressure of higher than 60MPa is applied before the gate shield is established, that is, before solidification after cooling, shrink marks can be removed(13)(14).

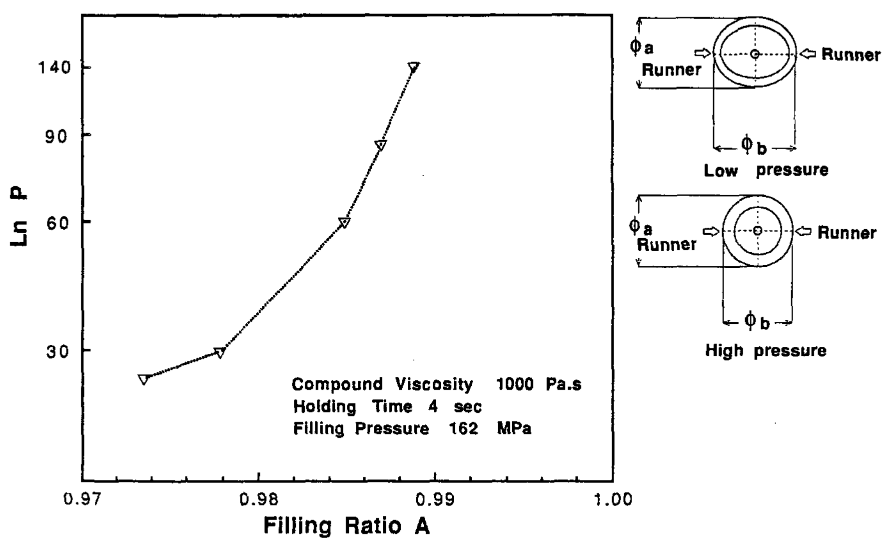


Figure 5. Relationship between the holding pressure and the filling ratio, A.

Figure 6 shows the relationship between the holding pressure and the percentage of incomplete mold ejection. As shown in the figure, when the pressure increases above 80MPa, the percentage of incomplete mold ejection greatly increases. This can be assured by the development of physical adsorption (namely vacuum conditions) between the surface of

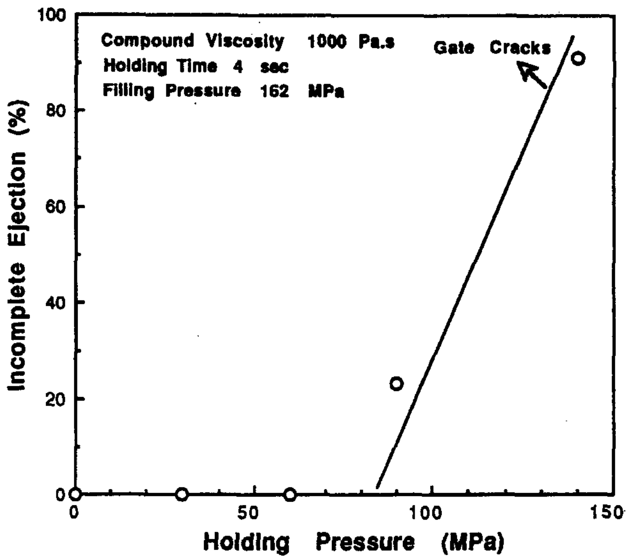


Figure 6. Relationship between the holding pressure and the percentage of incomplete mold ejection.

the sample and the metal die. Particularly, in the pressure region above 130MPa, the molded sample and metal die are firmly attached and cracks develop at sections of no adsorption, i.e., near the gate during ejection(15).

Next, we evaluate the relationship between the holding pressure P and the volumetric shrinkage ratio of $S_V=(V_{mold}-V)/V_{mold}$(16). From experiments, $V_{mold}=2.3481$, the hydrated weight and the dry weight of the sample are identical, and the sample is considered to be a compact body consisting of organic binder and ceramic powder. Then, the sample volume V can be obtained by the product of the weight M of the sample and the bulk density ρ, which was obtained by Archimedes's method(17)(18). Figure 7(a) shows the relationship between the holding pressures and S_V at various volumetric shrinkage ratios of the sample. Since a linear relationship holds between ℓn P and S_V in the region where P is higher than 60MPa, the relationship between P and S_V in the region can be represented by the following equation:

$$P=212.0e^{-179.8S_V} \quad (1)$$

Now, if we separate the shrinkage ratio in the ϕ and L directions, by making use of S_1 and S_2 which are obtained from the dimensional measurements of the molded samples, S_V can be defined as follows:

$$S_V=1-(1-S_1)^2\cdot(1-S_2) \quad (2)$$

Figure 7(b) shows the relationship between P and S_V, which was obtained by substituting S_1 and S_2 at various holding pressures into eq.(2). Since a linear relationship holds between ℓn P and S_V in the region where P is larger than 60MPa, the relationship between P and S_V can be represented in a manner similar to eq.(1) as follows:

$$P=275.2e^{-179.3S_V} \quad (3)$$

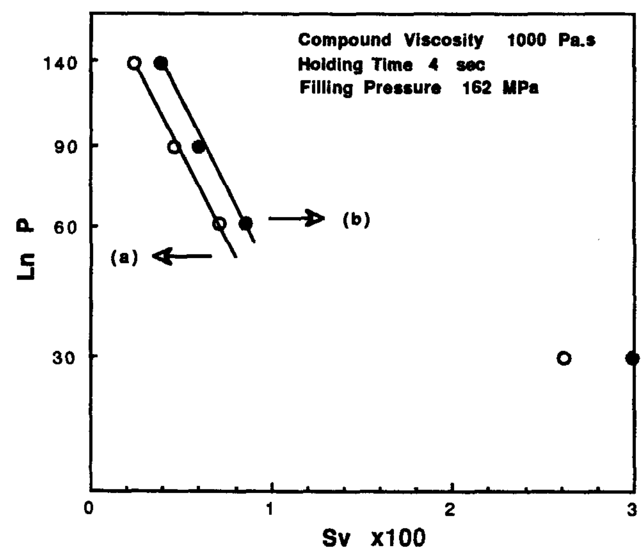


Figure 7. Relationship between the holding pressure and the volumetric shrinkage ratio.
(a) obtained by Archimedes's method
(b) obtained by measurement of the dimensions of the molded samples

When eq.(1) and eq.(3) are compared, the constants on the right-hand side of the two equations are very close to each other; thus, the relationship represented by eq.(2) holds between the S_V obtained by Archimedes's method and the results of S_1 and S_2 obtained by actual dimension measurements.

Also, by defining the relationship between ϕ and L as $\ell = V^{1/3}$, using the volume V of the molded sample obtained by Archimedes's method, the line shrinkage ratio, $S_\ell = (\ell_{mold} - \ell)/\ell_{mold}$, was obtained[16]. From the above measurement of dimensions, $\ell_{mold} = (2.3481)^{1/3}$, S_ℓ can be shown by the following equation:

$$S_\ell = 1-(V/2.3481)^{1/3} \qquad (4)$$

By substituting the values of V at various holding pressures, the relationship between ℓn P and S_ℓ can be obtained. The values of S_ℓ obtained by the above procedure are shown in Fig. 8 together with S_1 and S_2.

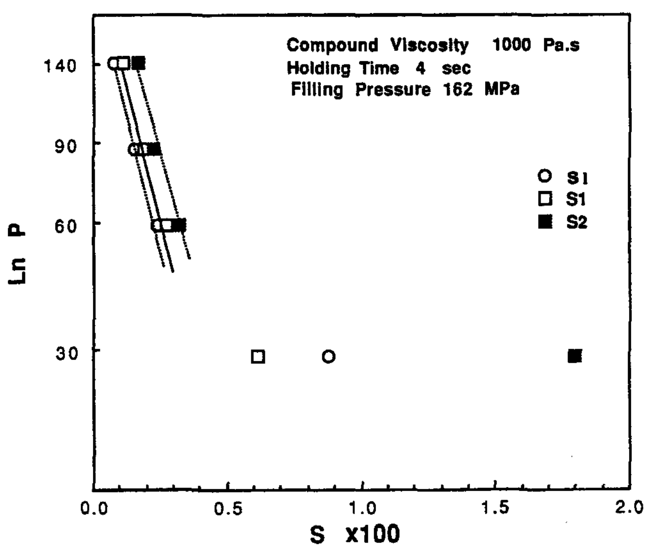


Figure 8. Relationship between the holding pressure and the line shrinkage S_ℓ obtained by Archimedes's method, and S_1 and S_2 obtained by measurement of the dimensions of the molded samples.

The figure shows that the value of S_ℓ in the high dwelling demonstrates that S_1 and S_2 are essentially equal. As explained, the bulk density ρ

can be obtained in terms of Archimedes's method from the weight M of the molded sample, and when the volume V of the sample is known, the molding shrinkage ratio, S_V and S_ℓ can be easily estimated.

Duration of holding pressure and viscosity of the compound: Figure 9 shows the relationship between the duration of holding pressure and the filling ratio of $A=(\phi_a/\phi_b)$ and percentage of incomplete ejection. Here, the viscosity of the compound and the holding pressure were kept constant.

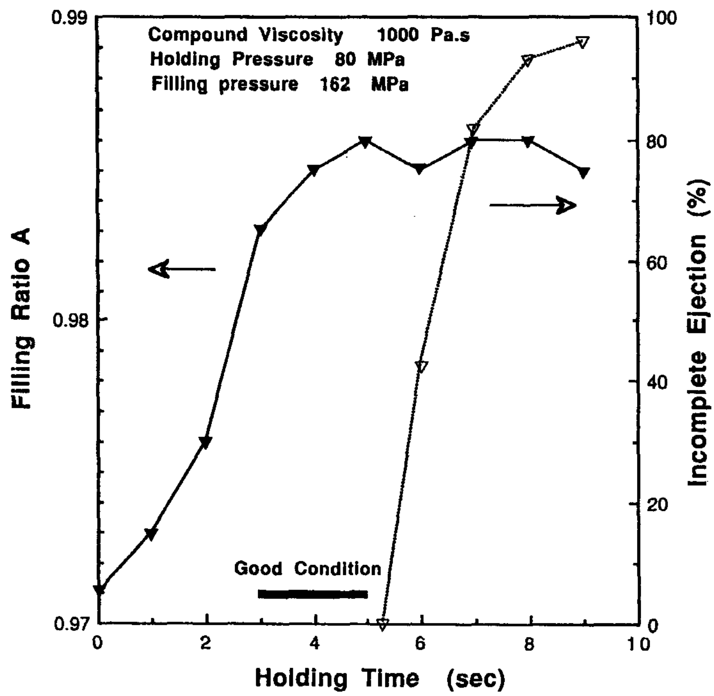


Figure 9. Relationship between the duration of holding pressure and the filling ratio, A, and the percentage of incomplete mold ejection.

As shown in the figure, as the duration of holding pressure increases, the value of A increases, and it reaches equilibrium after the duration of longer than 3 sec dwelling. However, when the duration of holding pressure is longer than 5 sec, the percentage of incomplete ejection increases greatly. This can be explained in the same way as in Fig. 6, where incomplete ejection developed in the high-dwelling region. With the increase in the duration of holding pressure, the air layer required for ejection disappears and physical attachment occurs at the interference between the surface of the molded sample and the metal die.

Figure 10 shows the relationship between the viscosity of compound and the filling ratio, $A=(\phi_a/\phi_b)$. Here, the duration of dwelling and holding pressure were kept constant. As shown in the figure, as the

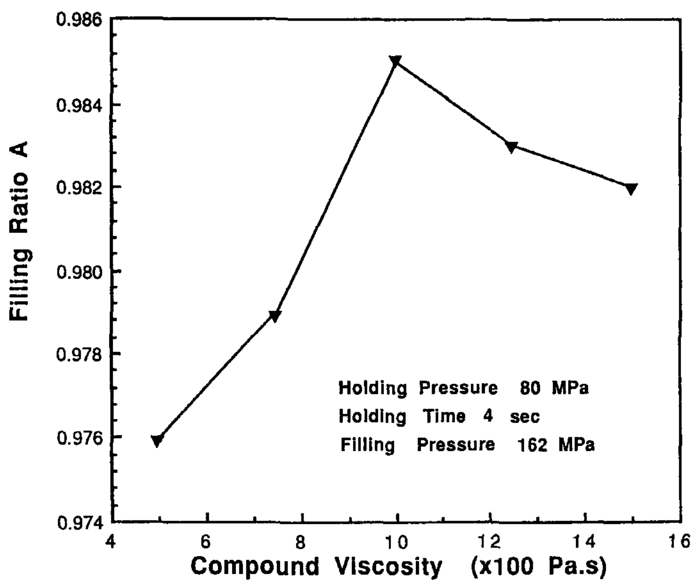


Figure 10. Relationship between the viscosity of the compound and the filling ratio, A.

viscosity of the compound increases, namely with the decrease of fluidity, the value of A increases, and reaches its peak at 1,000Pa.s. Then, it gradually decreases. This phenomenon can be explained as follows: the fluidity deteriorates greatly in the region of the compound whose viscosity is higher than 1,000Pa.s, and as a result, the succeeding compound does not completely fill the section left unfilled by the preceding compound by the time the molding is finished[13]. Considering the preceding findings, the optimum conditions for PSZ ferrule injection molding a holding pressure of higher than 60MPa but less than 80MPa, a dwelling time of between 3 sec and 5 sec, and a compound viscosity of 1,000Pa.s. Accordingly, in the present study, the dimensions of ferrules after sintering were evaluated for samples produced under the following conditions: a holding pressure of 80MPa, a dwelling time of 4 sec, and a compound viscosity of 1,000Pa.s.

Evaluation of Dimensions after Sintering

Table 1 shows the variability of ferrule hole diameters due to injection molding and sintering shrinkage. The standard deviation (SD) of injection molding of 0.66µm is excellent considering the SD of the metal dies, 0.52µm. The SD after sintered shrinkage is 0.82µm; although some processing is required to produce a final product, the molded samples conform with the near-net shape.

TABLE 1

Variability of ferrule hole diameters due to injection molding and sintering shrinkage.

Measured Point	Max	Min	Ave	SD
Pin Diameter 1/2L1	157.2	155.5	156.3	0.52
Hole Diameter 1/2L1 (Molded Sample)	156.3	154.1	155.4	0.66
Hole Diameter 1/2L2 (Sintered Sample)	125.3	122.5	123.8	0.82

(µm)

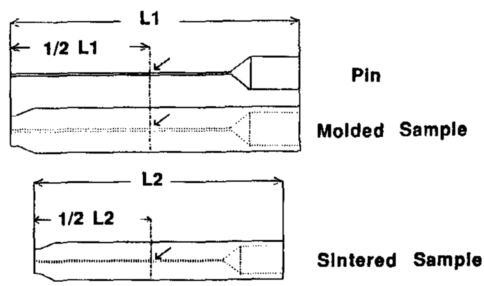


Figure 11 shows the surface roughness of the molding die and that of the sintered sample. The roughness of the die surface was 0.08µm Ra, and that of the sintered sample was 0.10µm Ra. Thus, there was no major difference between the die and the sintered sample, and transferability could be controlled within 0.10µm Ra. As can be assumed from the SEM photographs, the smoothness of the PSZ ferrule surface can be explained by the fact that the grains of PSZ ferrule after sintering are compact and are approximately 0.5µm in size.

Figure 12 shows the cylindricity measurement results of a PSZ ferrule. The average cylindricity was 6.0µm, and it faithfully transferred the cylindricity of the molding die, 2.0µm.

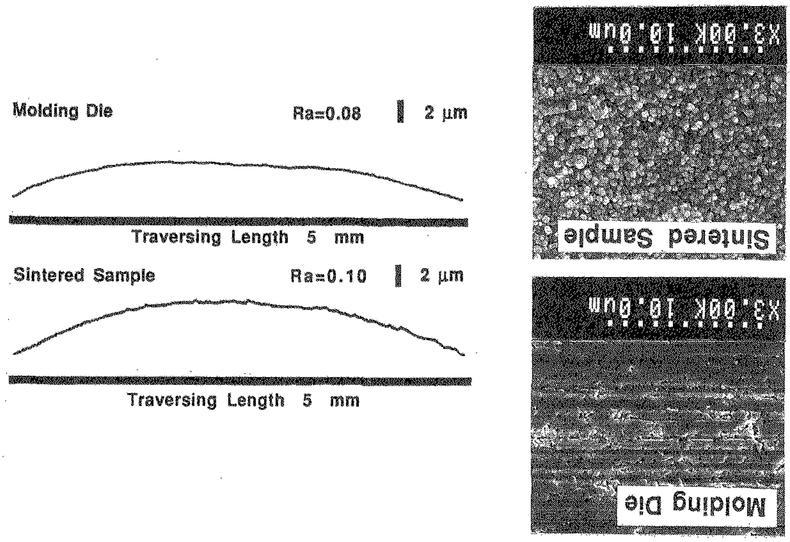
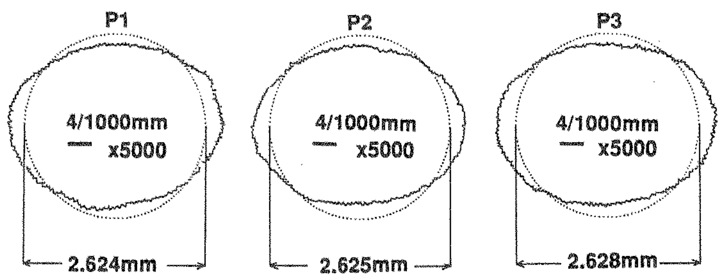
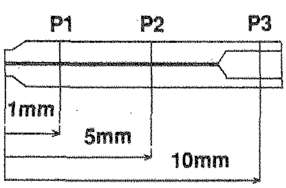


Figure 11. Surface roughness and SEM photograph of the molding die and of the sintered sample.





Cylindricity	P1	P2	P3	Ave
Sintered Sample	6.3	6.1	5.6	6.0
Molding Die	2.0	2.0	2.0	2.0 (µm)

Figure 12. Cylindricity measurement results of a PSZ ferrule.

The SD of ferrule hole diameters and the cylindricity measurement results were smaller than those in the case of filling ratio, A. We assume that the reason for this is that the filling ratio was measured immediately after molding, and the molded sample contained a great deal of residual stress and resulted in deformation(11). Furthermore, the debinding relieved the residual stress from the molded sample, and sintering enhanced the uniformness of shrinkage.

Table 2 shows the dimensional accuracy required for PSZ ferrules for SMF(1) and that obtained for the PSZ ferrules used in the present study. The former values indicate those required after grinding and polishing, and the latter indicate the values obtained from the sample after injection molding and sintering and before grinding and polishing. As is obvious from the table, all of the items in the table approximate the required dimensional accuracy, and thus the fabricated samples had a near-net shape. Namely, with the present fabrication procedure, complicated grinding and polishing as required for the conventional process can be avoided, and the cost can be reduced.

TABLE 2

Dimensional accuracy required for PSZ ferrules for SMFs and that obtained for the PSZ ferrules in the present study.

Measurement	Required Accuracy*		Injection Accuracy	
Outside Diameter	2499.0	(±0.5)	2593.0	(±6.0)
Eccentricity	0.7		5.8	
Cylindricity	0.5		6.0	
Surface Roughness	0.1		0.1	(μm)

Figure 13 shows the schematic diagrams of optical connector(a) and the PSZ ferrules produced by the conventional process (b) and by the present process (c). Both the surface conditions and shapes have the near-net shape.

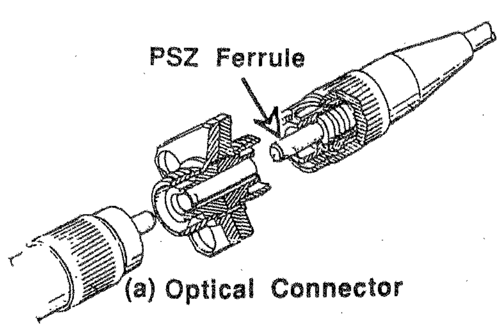


Figure 13. Schematic diagram of optical connector (a)

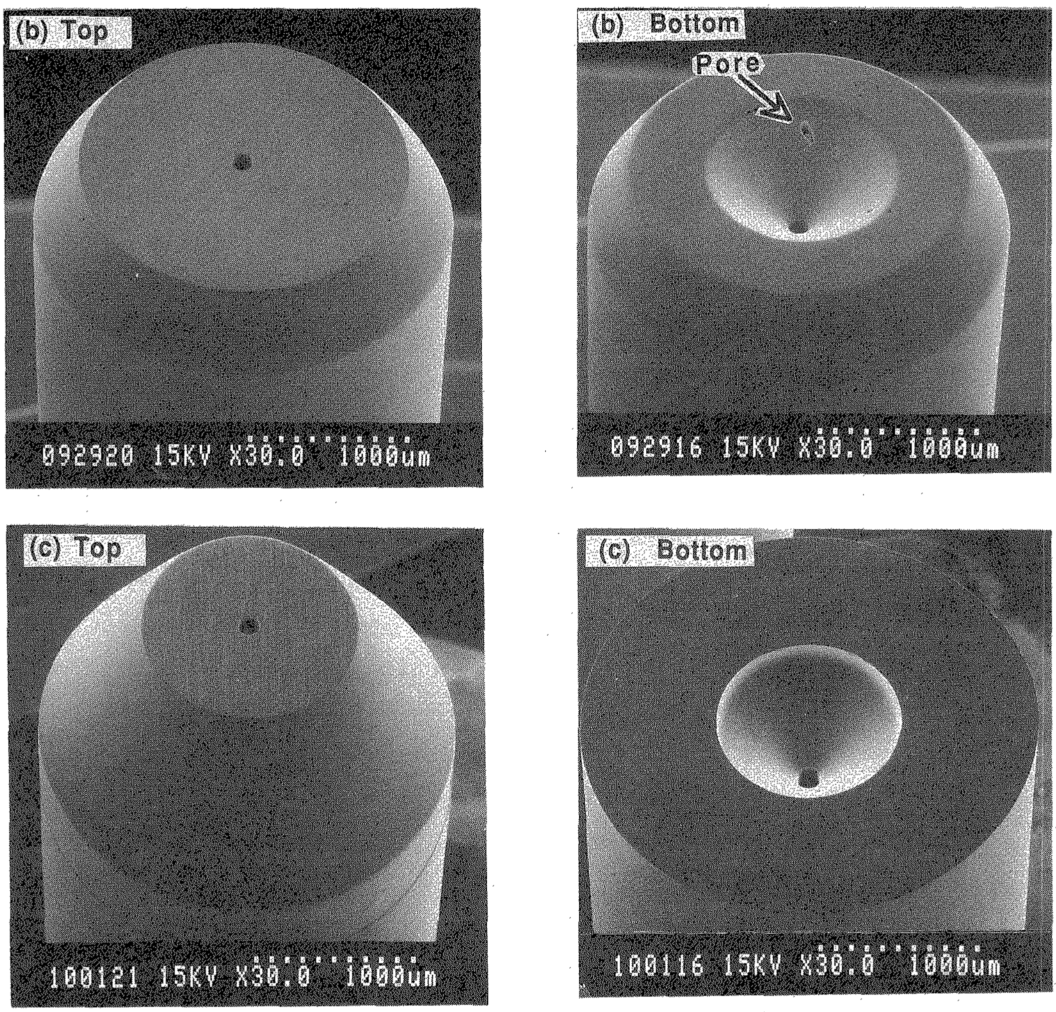


Figure 13. SEM photographs of PSZ ferrules obtained by the conventional process (b) and the sintered sample obtained by the present process (c).

CONCLUSION

In the fabrication of PSZ ferrules for optical connectors by the injection molding process, the molding die was optimized. Furthermore, the fabrication conditions of molding were evaluated in detail, and the dimensional characteristics of the sintered sample were evaluated. The results indicate that the ferrules produced by the present process can be used for practical purposes both in terms of material characteristics and cost. The following points summarize the results.

1) By using a molding die with an air vent and two gate balance runners, production of highly reliable PSZ ferrules (particularly in terms of material characteristics) has been accomplished.
2) From the viewpoint of mold shrinkage ratio, filling ratio and percentage of incomplete ejection, the optimal conditions for fabrication of injection molding samples are: a holding pressure of higher than 60MPa and less than 80MPa, a duration of dwelling of longer than 3 sec and less than 5 sec, and a compound viscosity of 1,000Pa.s.
3) From the weight of the molded sample, the bulk density was obtained by Archimedes's method. Furthermore, by obtaining the volume of the molded sample, it was found that the molding shrinkage ratio S_V and S_ℓ can be easily estimated.
4) The PSZ ferrules for SMF fabricated under the conditions listed in point 2) approximate the dimensional accuracy requirement and demonstrate a fairly high level of the near-net shape characteristics.
5) By the use of the injection molding process for the fabrication of PSZ ferrules, complicated grinding and polishing processes have been eliminated; this production process thus lends itself to mass production and cost reduction.

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