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Near-net-shape forming of zirconia optical sleeves by ceramics injection molding

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Abstract

Conventional sleeve blanks are currently produced by extrusion, which causes large dimension variations. Consequently it takes longer time and higher cost in the polishing/lapping process to obtain the final dimensions. An injection molding process has been developed for the production of near-net-shape ceramic sleeves. The special designed injection mold has been fabricated to produce sleeve blanks with accurate dimensions. With our zirconia/binder system, the overall debinding/sintering process time is about 26 h for each batch and this provides economic efficiency in mass production. The influence of using recycled feedstock on green body dimensions is investigated thoroughly in this study. By optimizing the process parameters in the molding and debinding stages, one could not only control the dimensional tolerance up to 10 μm (diameter control $<0.4\%$ with $>99\%$ yield) in blank sleeves but also obtain sintered sleeves with good cylindrical shape. The developed ceramic injection molding process has great advantage in reducing machining time up to 60% in the subsequent grinding/lapping process.

Keywords: A. Injection molding; Sleeve; Near-net-shape; Tooling; Adapter

1. Introduction

In the present fibre-optic transmission system, a number of optical connectors are required. The main function of these interconnection systems is to link the cable head and the terminal equipment or to allow the connection of multi-fiber cables. Such connections may be attached to the equipment bays or take the form of patch cords for a possible optic-optic distribution frame. Essential parts of these systems are the ferrules and alignment sleeves, which are mainly made of zirconia ceramics. Two ferrules are used in such a way that the fiber ends can be positioned opposite each other repeatedly with a slit ceramic sleeve. The most critical tolerance is the lateral offset between two fibers. Thus, manufacturing zirconia sleeves will emphasize the dimension control on the inner/outer diameters and also the concentricity. Fabrication of zirconia sleeves involves a series of processes from powder mixing to final grinding/lapping. The most important goal is obtaining a near-net-shape sintered body which can greatly reduce the subsequent machining cost. The other requirements such as mechanical strength, surface roughness and microstructure of the sleeve can be achieved by proper choice of zirconia powder and processing parameters.

Ceramic fabrication by injection molding offers automatic production with minimal expensive secondary operation. It involves compounding the fine ceramic powder with a blend of polymers or wax so that the feedstock can fill the die cavity during the injection stage. The organic binders embedded in the molded green parts have to be subsequently removed via thermal pyrolysis or solvent detracting prior to sintering. By proper controlling the process parameters, it is possible to obtain a near-net-shape blank, which can minimize grinding and hence the machining cost.

A number of workers have investigated the properties of powder formulations. With and Witbreuk [11] have investigated the mechanical properties and microstructure of Y-TZP ceramic under various controlled parameters. Nogueira et al. [9] have shown the influence of different ceramic powder sizes on the various stages of the process. Edirisinghe and Evans [7,12] have studied the flow properties of different ceramic injection formulations.

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Tseng and coworkers [1,2], have investigated the influence of powder formula and processing parameters on microstructure of molded ceramic samples. Sakai [13] has successfully utilized the ceramic injection molding in manufacturing of "finger" as a copying machine part. In addition, Tseng [5] has shown the influence of molding parameters on warpage of molded bar samples. However, no studies have been addressed to manufacturing ceramic sleeves. Recently, plastic sleeves have been studied due to the demand of cost reduction. Thus, Sato et.al. [14] adopted a thermosetting injection molding process to produce plastic sleeves with an average retention force of 506 g. However, the long-term reliability of such sleeves is still under investigation.

In this study, a low-molecular-weight organic binder system containing paraffin wax and polyolefin-type polymer was used for molding ceramic sleeve blanks. This binder system was originally developed by Liu and Tseng [1,2,4] and we used binder formula directly from their experiments. A conventional 20-ton injection machine was used in molding the sleeves. A three-plate mold with four die cavities was designed to achieve the high accuracy of the molded sleeves. Since the experimental results are used as a preliminary study for mass production, focus was made in developing a reliable mold and in controlling final sleeve dimensions to tight tolerance. Meanwhile, effects of recycled ceramic feedstock and degating methods on the sleeve dimensions are also investigated in this study. The molded sleeve blanks when subjected to subsequent grinding process show better machining efficiency in comparison with the conventional extruded sleeves.

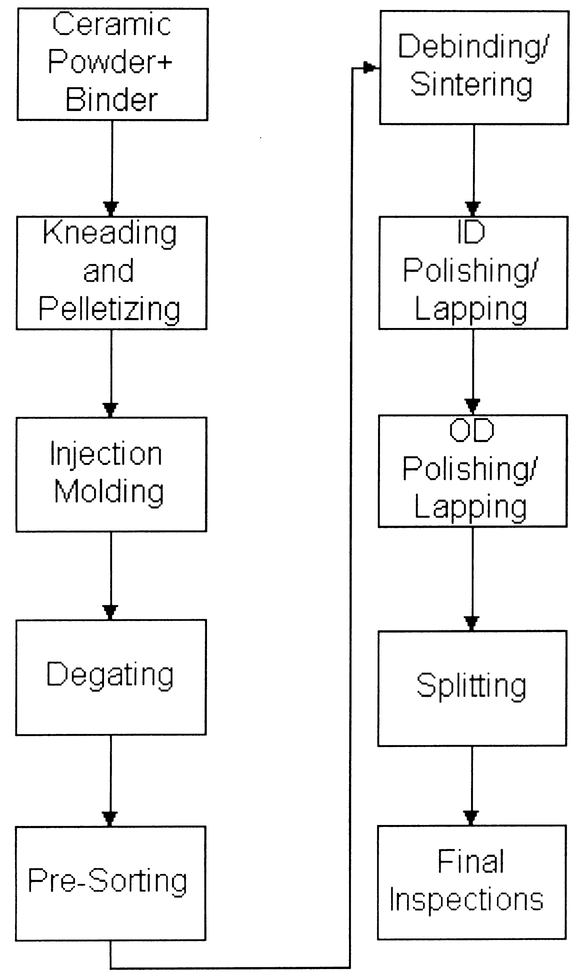

Fig. 1. The manufacturing process of ceramic sleeves. The molding and lapping processes are all included in this flow chart. The illustration shows the case that degating is performed before debinding process.

2. Experimental procedure

The manufacturing process of ceramic sleeves used in our experiment is shown in Fig. 1. A centerless grinding machine[1] was used for fine grinding the outer diameter (OD) of blank sleeves. A specially designed lapping machine [18] was used for fine grinding the inner diameter (ID) of blank sleeves. Since the tolerance of sleeve ID has to be controlled within 5 μm, a smaller material removal speed (~1 μm) is necessary in the ID lapping process. Therefore, the ID lapping is the most time-consuming stage of the production process.

2.1. Ceramic feedstock preparation

A commercially available YTZ ceramic powder, containing 3 mol% Y_2O_3[2] with an average particle size of 0.6 μm, was used in this study. The organic binder consisted of paraffin wax, vinyl acetate polymer,[3] and stearic acid.[4] The volume fraction of powder (i.e. solid loading) in the molding mix is fixed at 55% which is a reasonable amount. The paraffin wax was mixed with polypropylene in 60:40 weight ratio and stearic acid was mixed in 3% of total weight. The content of stearic acid affects the viscosity and pore size of binder system as revealed by Tseng's work [1]. Such an organic binder system has been established and tested in our molding experiments for zirconia ferrules. This binder system provides good injection molding capability and the flexural strength of sintered samples reached 700 MPa to allow machining in latter process [15]. To determine the temperature ramp in the debinding process, performing thermogravimetric analysis (TGA) of the

[1] SKS N250, Micron, Japan.
[2] HSY3.0, Daiichi Kigenso Kagaku, Japan.
[3] Gin-Ming Chemicals, Taiwan.
[4] Reagent grade, Showa Chemicals Inc, Japan.

binder is necessary. Here we took the results from Liu's work (Fig. 1 in [3], 55% of solid loading) and adopted an optimized debinding and sintering profile.

2.2. Mixing and compounding

Each zirconia compound and binders were first kneaded together in a high shear extruder.[5] The temperature control was set to be 70–120–140–160–180°C from barrel to nozzle at 1500 rpm for 5 min. The extruded pellets are in cylindrical shape with 3 mm in diameter and 2–4 mm in length. The fully plasticized feedstock was tested in the capillary rheometer[6] with a capillary 1 mm in diameter and 10 mm long to measure the suspension viscosity at a temperature range of 65–85°C. The relationship between shear rate and viscosity was used as a reference for the subsequent molding process.

2.3. Injection molding

The injection-molding machine used in this study was the Battenfield BA200, a screw plastification in-line type with four heating zones. Due to the abrasive nature of ceramic material, SKD61 based steel was used in the nozzle seat and the screw coated with Ni-Fe was also used in the molding machine. The molding conditions are shown in Table 1. Each molded green sleeve weighed 0.4 g while the runner weighed approximately 11 g.

The development of cracking or voiding in ceramic injection molding is related to non-uniform shrinkage [6]. The holding pressure determines the pressure in the mold cavity at the time when sprue solidifies and hence determines the resistance to shrinkage in the cavity. Thus, the holding pressure must be adjusted if there is a problem in the volume shrinkage. A higher mold temperature was considered to increase the internal stress, thus, the mold was controlled in room temperature during operation [5,8].

2.4. Degating

Degating can be made either before the debinding/sintering or after that. An automatic degating system is applied if degating is processed before firing. Chosen of degating method can greatly affect the longitudinal dimension as well as the quality of sleeves. After a series of experiments, we used a special designed cutting drill operated in a proper spindle speed to remove the gate portion and also to form a fillet on the sharp edge. The drilling module in this system was designed with a tunable descent speed, which was controlled by the pneumatic slow descent mechanism. Thus, the cutting surface can be maintained in a good quality without further polishing.

Table 1
Molding parameters used in this study. The screw size used in the CIM is 22 mm in diameter

Temperature (°C)	
Mold on both sides	25
Nozzle	155
Cylinder	150–155–140
Pressure (bar)	
Injection	61–40–5
Packaging	35–33
Time (s)	
Filling	0.2
Packaging	0.35–0.27
Cooling	8
Cycle	20
Screw speed (%)	10–8–6

A special fixture holding ten sleeve blanks is also designed when degating is made after firing. A diamond wheel with 0.7 mm thickness was used as a cutter to furnish the gate portion. A faster feeding can result in edge deformation and thus one should be aware of this side effect during grinding process. Comparison of two methods will be discussed in the result section.

2.5. Debinding and sintering

The next step, binder removal, was done in a chamber furnace.[7] The process of thermal debinding in the air at atmospheric pressure was used. The air in the furnace was constantly renewed at a rate of a few liters per hour. The heating ramp, used for binder removal, basically follows the two stages outlined by [1]: (1) The oxidative degradation of the binder is in the surface region. (2) The evaporation of low molecular weight components of the blend is at the suspension-gas interface. The first stage occurs in the temperature range of 250–300°C and the second, 350–400°C. The sintering process followed immediately the debinding process. The debinded sleeves were sintered by heating to 1500°C and by soaking at this temperature for an hour in the same chamber furnace. The total debinding and sintering time was about 26 h. The density of the sintered samples was measured using the water immersion method and found to be >5.9 g/cm^3 in average. It is about the same density as the one used in the commercially available ceramic ferrules.

3. Mold layout

Design of injection mold plays a major role in ceramic fabrication. Many of the basic molding parameters for

[5] Model 70-20 VEX-6, KCK Industries Co., Japan.
[6] Model CFT500, Shimadzu Co., Japan.
[7] Model BF51524C, Lindberg, USA.

ceramics are similar to those for polymer. However, there are two biggest differences between molding polymers and ceramic mixes: elasticity and thermal conductivity [10]. Ceramic mixes are much less elastic than polymeric ones and they have a much higher thermal conductivity. Lower elasticity means that ceramics deformed less after being stressed, such as when they flow through a gate before entering a mold. The higher thermal conductivity of the ceramic mixes means that they cool much faster than polymer does. This results in the difficulty of mold filling process. Thus, the most critical areas in designing the mold are mold layout, gating system, cooling system, and removal of the parts from the mold.

There are several things need to be aware in design these injection molds:

1. All runner paths need to be coated with Ti before mass production. In the pin coating process, one needs to be aware of the lateral deformation of the pin. Uncontrolled thermal deformation of heat-treated inserts will result in the deviation of concentricity of molded sleeves.
2. There are two types of mold designs in manufacturing ceramic sleeves: two-plate mold and three-plate molds. We have used both designs in our laboratory. The two-plate mold was designed with hot-runner system and the other was applied with runner locker pins. When the hot runner system is applied in the two-plate mold, a temperature control (i.e. insulation) of the mold is critical to ensure the operation of molding since the temperature on both sides is extremely different. This paper will only address the problem in the three-plate mold.
3. A self-locking with auto-centering mechanism is necessary in the tool design since the concentricity of sleeve is important to the polishing process. This mechanism will enable the assembling accuracy of core parts each time when we maintain the tool.

3.1. Gate system

A round runner with diameter of 5.0 mm was used in this design. The fan-gate type with circumferential thickness of 0.85 mm was located on one end of the cavity. Gate position can have an important meaning on whether or not welds will occur. In our single gated mold, weld can be avoided by side-gating the cavity to generate plug flow [6]. The residual stresses in conventional molding are raised, as the holding pressure is increased [8]. Here, we adapted a larger diameter in sprues and gates so that sufficient pressure is maintained in the cavity to inhibit the nucleation of shrinkage voids without using high levels of hold pressure.

The distance between gate and runner end has been optimized through our experimental study so there is a breaking near the gate land. The diameter of the gate land (i.e. located on the surface of fan-gate) has set to be 0.8, 1.0, 1.2 and 1.4 mm for trial. With a choice of 1.2 mm, the molded sleeves can be ejected smoothly at the end of each shot. Considerable time was spent in these experiments due to lack of mathematical modeling.

3.2. Cooling system

The mold temperature was kept at 25°C. Special care was made in the locating ring area due to the inlet of hot slurry. The cooling loop has been placed around the cavity with a distance of 10cm to the cavity center.

3.3. Green parts removal

A three-plate mold was used in this study, therefore, the green parts was automatically separated from the runner which can be ejected by the plate on the fixed side. The runner lock pin has to be short enough in order to avoid the runner/gate break before parts were removed from die cavities. There is a taper design in the cavities (0.2-degree), so the part was easily ejected and the concentricity can be kept good after the sintering process.

4. Results and discussion

4.1. Molded sleeve blanks

The dimensional accuracy of the as sintered and finally finished sleeves was evaluated for the fabricated samples. The dimensions for injection molded sleeves and commercially available extruded sleeves are listed in Table 2. The geometric dimensions include inner and outer diameters, length, cylindricity, and concentricity. The first two items have directly impact on the machining time; thus near-net-shape condition is the objective in this study. The concentricity needs to be controlled within 20 μm so the grinding/lapping process can be performed without shortening the tool life. Length and concentricity are primarily controlled by the cavity dimension and tooling accuracy. The concentricity is less affected by the molding parameters as we experienced (not shown). Once the auto-centering mechanism is included in the tool, the concentricity can be reduced from 0.05 mm down to 0.015 mm (i.e.2 σ value). Cylindricity can not be achieved either by tooling or material composition, so an alternative process need to be taken. Effects of solid loading and binder formulation on the dimensions and warping were significant and have been studied by Tseng's work through analysis of variance (ANOVA) [5]. Here, we will not repeat those experiments

Table 2
The dimension precision and properties comparison of commercially available extruded and molded sleeve blanks. The extruded sleeves are adopted from TOSOH Corporation, Japan [16]. The commercial sleeve specifications (i.e., after grinding/lapping) are from Bellcore standard [17]

Items	Measurements	Extruded sleeve blanks	Molded sleeve blanks	Commercial sleeve specifications
Pulling force	NA	NA	NA	<450 g
Length	Calliper	11.4±0.05 mm	11.35±0.05 mm	11.4±0.2 mm
Outer diameter	MS/Micrometer	3.5±0.02 mm	3.40±0.005 mm	3.2±0.02 mm
Inner diameter	MS	2.46±0.02 mm	2.48±0.005 mm	$2.491^{+0.005}_{-0}$ mm
Concentricity	MS/Micrometer	<0.03 mm	<0.015 mm	NA
Cylindricity	MS/Micrometer	<0.01 mm	<0.015 mm	NA
Insertion loss	Optic power meter	NA	NA	<0.2 dB

in the sleeve production. By fixing the material formulation, influences of continuous production, recycled material, degating methods on sleeve diameters are involved in this study.

Batch testing is necessary to ensure the reproducibility of measured results. Figs. 2 and 4 show the experimental study for sample size of 60 pcs under a continuous batch operation. The IDs of sintered sleeves are precisely controlled under tolerance of ±0.005 mm with yield of 99% (i.e. 3 σ value). A similar tolerance was obtained in OD. These data are measured on non-gate side and center portion of sleeves. On the gate side, the holding pressure dominates this region due to solidification delay, a smaller shrinkage is therefore occurs and the OD becomes larger (e.g. 0.04 mm larger as we see in Fig. 2). Such a horn-type deformation greatly affected the quality of final sleeves because of the difficulty of holding sleeves' OD while grinding ID. Improvement of horn-type deformation is necessary, as we will discuss later.

The shrinkage ratio of sintered sleeve blanks is computed as 1-(blank dimension)/(cavity dimension) and is about 20.76% in radial direction. The longitudinal dimension (i.e. length) is mainly controlled by the degating system and can be easily obtained within the tolerance of ±0.2 mm, which is required by the commercial specification (Table 2). The shrinkage ratio in this longitudinal direction is about 20.58%, which is about the same as what we got in radial direction.

Effects of re-used material in sleeve dimensions become critical in mass production, since weights of sprue and runner account for 87% of total material usage. Thus, investigation of re-used material is interesting to the researchers. Here, we used a batch of 5 kg ceramic material and recycled runner and sprue for five times. The recycled material has less influence in the slopes as we see in the shear rate vs. viscosity diagrams (Fig. 3 measured by CFT-500D rheometer, Japan). It means that the fluidity has small variations in molding so the

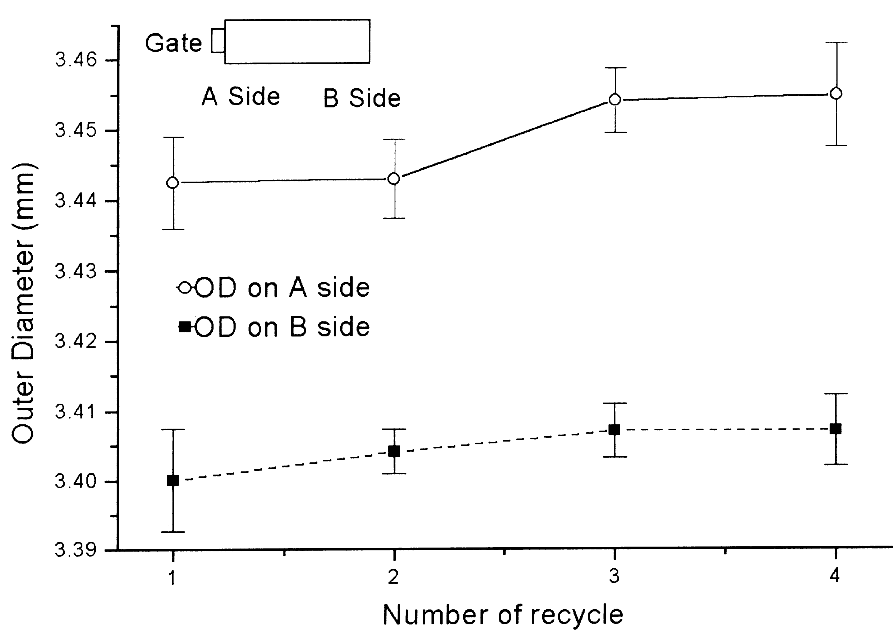

Fig. 2. Outer dimensions on both sides measured from four continuous batch operations. There are 60 samples measured in each cycle. Error bars indicate 3σ value in all figures. (σ is the standard deviation).

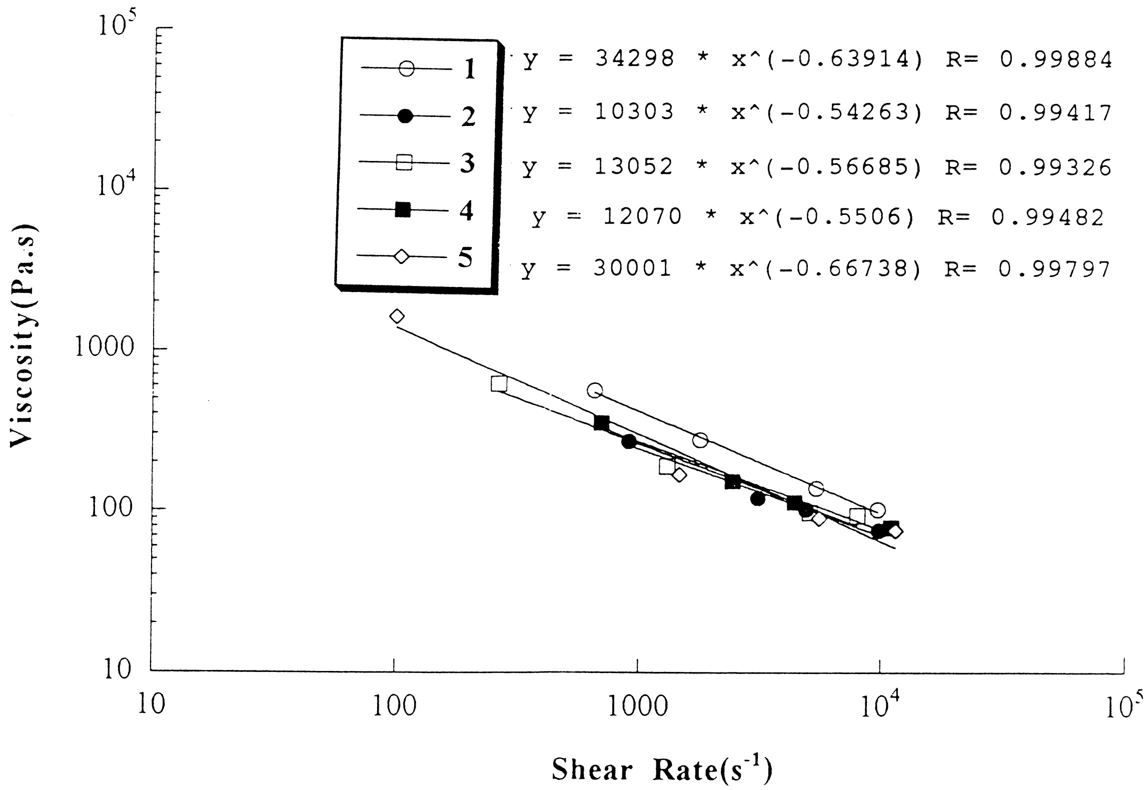

Fig. 3. Shear rate vs. viscosity curves for the number of recycled time. The solid loading is 55% at the first-run.

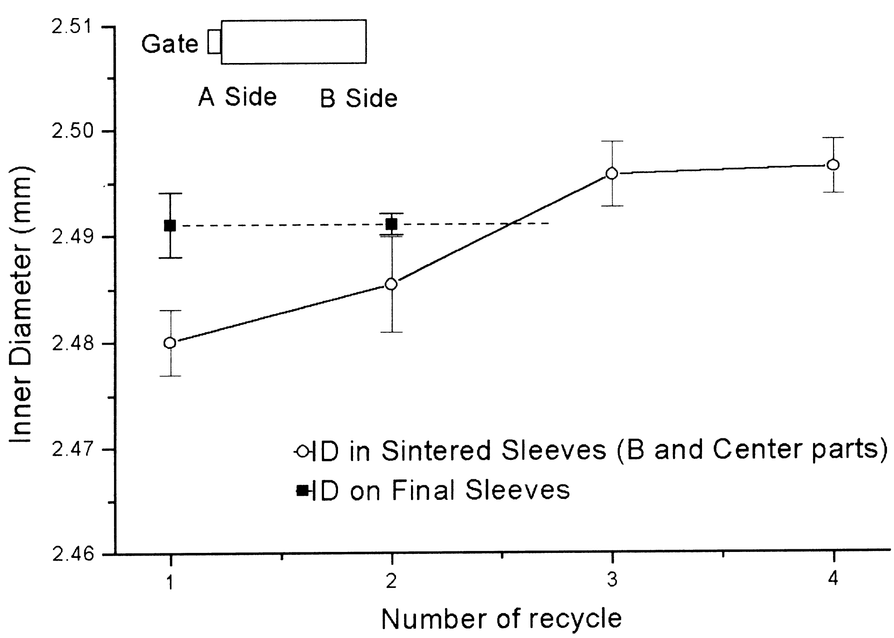

Fig. 4. Inner dimensions measured from sintered sleeves during four continuous batch operations. The dotted line represents the final ID of sleeves after lapping/polishing process.

same molding parameters can be applied each time. However, the conjunction between fan-gate and sleeve become fragile as we observed in the forth and fifth molding. It can be reasonably considered that the poor-strength results from the degradation of recycled material and also from the evaporation of organic binder, such as polyolefin-type polymer at high temperature [4]. Such a fragile behavior may result in the difficulty of sleeve ejection in later automation production. Fig. 4 shows the sleeve IDs at the first four runs. As we see in this figure, the averaged IDs of blank sleeves are 2.480, 2.485, 2.496, and 2.496 mm for the first four runs respectively. If one considers the final sleeve ID as 2.492 mm, which produces the pulling force in the range of 300–450 g [see Fig. 7 (a)], the first two runs thus provide sufficient dimension allowance for lapping/grinding. The machining time is significantly reduced and manufacturing cost is low in near-net-shape molding. The third and forth batches were not polished since the IDs were out of specifications. Experimental results show that the re-cycled can be used for two times at least without loss of any ID accuracy.

Effects of recycled batches on ODs are depicted in Fig. 2. The ODs have less variation in comparison with

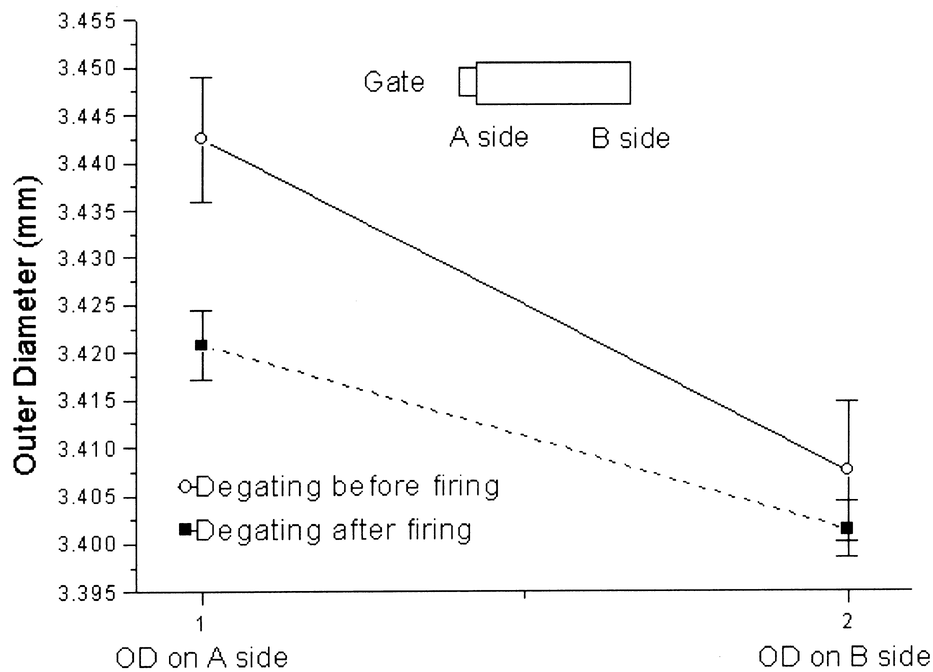

Fig. 5. Measurements of outer diameters on both sides of sintered sleeve. It shows that degating after sintered produces better cylindricity.

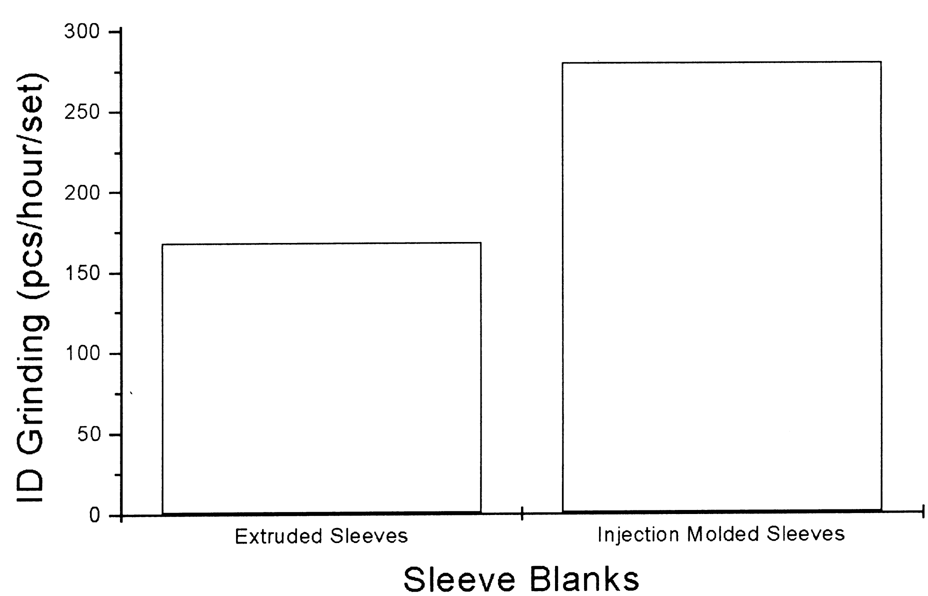

Fig. 6. Comparison of ID production capability by using extruded and injection molded sleeve blanks. Apparently, the near-net-shaped sleeve blanks have impact on the increasing production efficiency during the mass production.

IDs as the number of re-cycled time is increased when we measured from non-gate side. The ID seems to be more sensitive to the number of re-cycled time. The core pins designed in the cavities are solely fixed on both sides without temperature control (e.g. cooling pipe) inside. Such mold layout was thought to produce a non-uniform temperature distribution, which in turn, would result in an unbalance density distribution over the cross-section of the sleeves, and might produce a larger shrinkage ratio around inner diameter. There are two methods to overcome this problem: (1) increase the injection speed, and (2) keep a uniform temperature distribution within the die cavities. Increasing the injection speed will result in the increase of internal stress, which may cause the dimension instability. Embedding a cool pipe in the core pins will be a good try for future study. Excluding the pin portion, we are able to obtain a precision molded sleeve within 10 μm tolerance (Fig. 2) and thus re-cycled material can still be used for many times depending on applications.

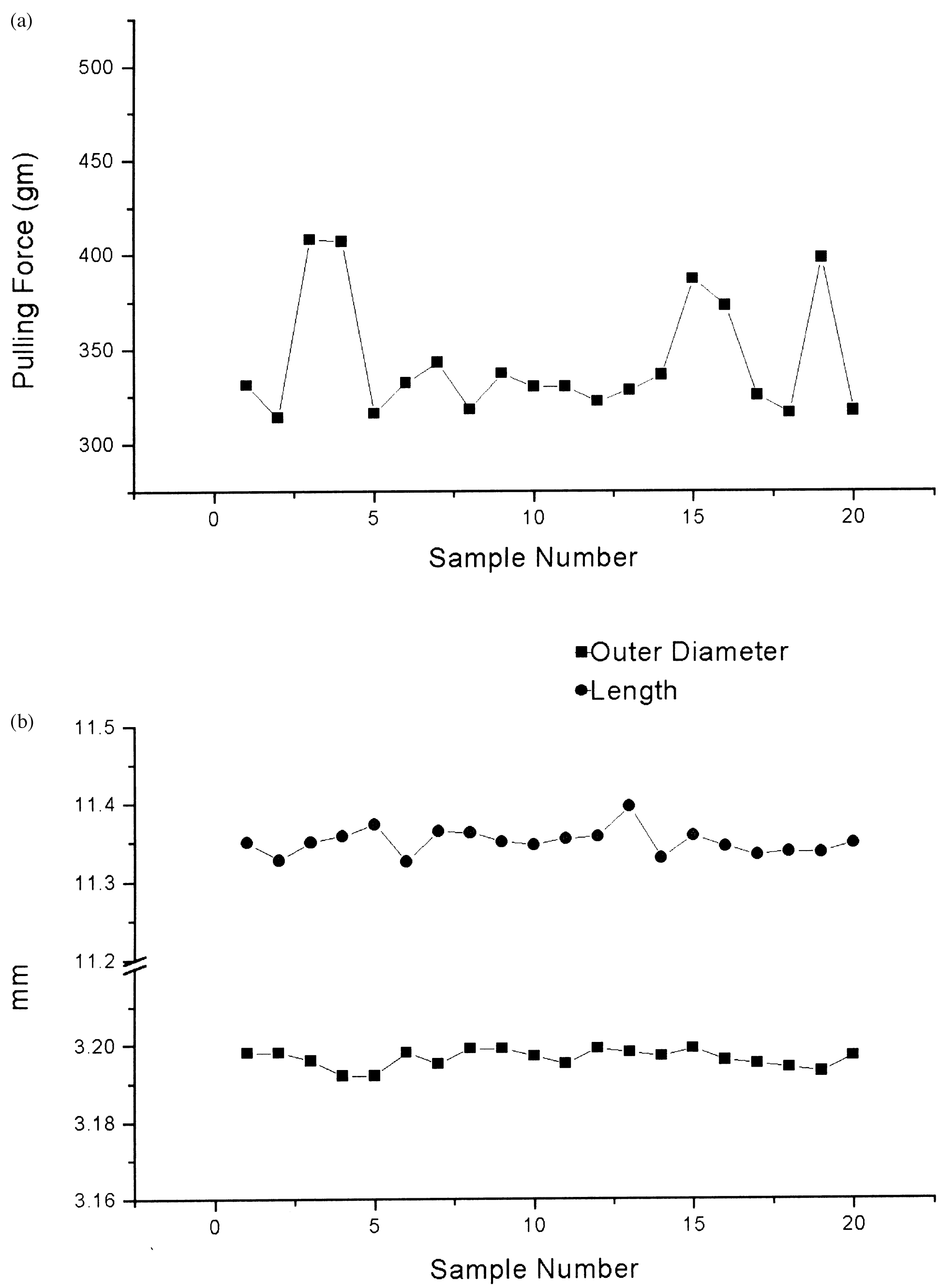

Fig. 7. Dimensions measured from continuous production of 20 polished sleeves. The sleeve blanks were obtained from our injection-molded process. (a) Pulling force measurements (b) Outer diameter and length dimension measurements.

If we assume that the length/shrinkage ratio is related to volume/shrinkage ratio by a cubic power, it thus can be expressed as:

$$[1 - (\text{blank dimension}/\text{cavity dimension})]^3 \propto [1 - \text{solid loading}] \quad (1)$$

Since the ID's shrinkage ratios for the four re-cycled materials are 20.76, 20.60, 20.25, 20 and 25% respectively, the solid loading at each batch will be increased slightly. There is no addition of binder component at each recycled material so the only losses are binder wax/polymer. Relatively, the recycled material slightly increases the solid loading of material. In view of the experimental results (Fig. 4 and the lower curve in Fig. 2) the dimensional variations depend not only on the solid loading of material, but also on the temperature distribution within cavities.

In the molded sleeve, shrinkage ratio varies from gate side to non-gate side. The deformation is enlarged after the debinding/sintering process. Here we utilize the fan-gate structure to provide the radial constrain near the gate side. In other words, we alternate the degating process and leave the gate with green sleeve in the debinding/sintering process. Effects of alternating degating process on cylindricity are shown in Fig. 5. Here we applied an automatic degating system for green sleeves. A diamond wheel was used to cut 10 sintered sleeves at a time after they were sintered. Apparently, the later produced better cylindricity and the dimension variation can be reduced from 30 to 15 μm. Thus, the fan-gate provides a certain constraint to the shrinkage of sleeve during the debinding/sintering process. Further improvement of cylindricity can be made by increasing the length of green sleeve for lateral precision cutting in the post process.

4.2. Final finished sleeves

A series of fine polishing processes was applied in our molded blank sleeves in order to get final finished sleeves. The grinding process starts from ID grinding where all sleeves are clamped through a special fixture on ODs. After obtaining the required ID dimensions, 60 sleeves were casted for OD grinding. The same casted sleeves were then split with a diamond saw to get the final product. All the processing parameters have been optimized before the test run and they are not included in this paper.

The production efficiency of ID grinding on extruded and molded blank sleeves is shown in Fig. 6. The most time-consuming ID grinding process was limited by 168 pcs/h/set when we used extruded sleeve blanks [18]. The production efficiency is increased up to 280 pcs/h/set when our molded sleeve blanks were used. The loading and unloading time is not included in this case. Such an improved efficiency is substantially beneficial from the near-net-shaped sleeve blanks obtained from our injection molded process. Fig. 7 (a) shows the pulling force measured from 20 lapped sleeves. They are all in the desired specifications. The pulling force is directly related to the IDs of polished sleeves so measurement of inner diameters is omitted in data presentation. Normally, polished IDs which are in the range of 2.491–2.493 mm produce the pull force in the range of 300–450 g. Fig. 7(b) shows the outer diameters and length of sleeves. They fulfill commercial specifications as depicted in Table 2.

5. Conclusions

We have developed a three-plate tool used for injection molding zirconia optical sleeves. Comparing results from extruded and molded sleeve blanks, we were able to produce near-net-shaped sleeve blanks, which greatly reduce the machining time up to 60% in the lapping/polishing process. The injection mold provides many advantages to the near-net-shape forming, such as dimension stability, short cycle-time, and geometric accuracy.

In the review of our study, the inner and outer diameter can be well controlled within 10 μm. The outer diameters of sleeves can still be controlled within 10 μm when material was re-used for four times. This provides a sufficient commercial benefit for the reduction of the manufacturing cost. The sleeve dimensions depend not only on the number of recycle time (i.e. solid loading) but also on the condition of solidification in the cavities.

The geometric constraint provided by fan-gate design can improve the cylindricity up to 50%. Thus degating processes have been made after the debinding/sintering cycle in order to maintain a good cylindricity for later polishing process. The production efficiency obtained in the lapping/grinding process has confirmed the feasibility of our precision molded sleeves in future commercial mass production.

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