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The processing-related fracture resistance and reliability of root dental posts made from Y-TZP

Aleš Dakskobler[a,*], Peter Jevnikar[b], Čedomir Oblak[b], Tomaž Kosmač[a]

[a] *Jozef Stefan Institute, Engineering Ceramics Department, Ljubljana, Slovenia*
[b] *Medical Faculty, Cathedra for Dental Prosthetics, Ljubljana, Slovenia*

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Abstract

In this study we have looked at how the processing affects the mechanical properties and reliability of root dental posts (RDPs) with a core that are made of yttria partially stabilized tetragonal zirconia (Y-TZP). These RDPs were designed for the aesthetic restoration of endodontically treated teeth. Low-pressure powder injection molding (LPIM) was used to wet form the green parts. The production process was found to have a significant effect on the mechanical properties and the Weibull modulus of the sintered specimens. During the shaping process different types of defects are introduced. These defects have a major effect on the reliability of the sintered specimens. A microstructural analysis of the sintered specimens was carried out to map these characteristic defects. With an appropriate adjustment of the molding parameters it was possible to eliminate most of these defects, and better reliability was achieved.

In addition, the effect of HIPing the sintered specimens was investigated. The results showed the importance of this post-sintering processing step on the reliability and fracture strength of the prepared parts.

Keywords: Injection moulding; ZrO_2; Mechanical properties; Biomedical applications

1. Introduction

Yttria-stabilized tetragonal zirconia (Y-TZP) has become increasingly popular as an alternative high-toughness core material in dental restorations because of its biocompatibility, attractive mechanical properties and its superior natural appearance compared with metal dental restorations.[1] Such attributes have resulted in Y-TZP being used for all-ceramic bridges, implant suprastructures and root dental posts (RDPs).[2] These posts are used when there is an insufficient volume of natural dentine to restore the dental crown to its natural shape, shade and function with a fixed prosthetic appliance or restoration, called a prosthetic crown.[3,4]

Recently, a novel RDP with a so-called retentive core was designed.[5] This core can be adjusted to a suitable form for each individual case by adding suitably translucent porcelain, or it can be easily reshaped by grinding with dental burs. In order to minimize the volume of material to be removed by grinding, the cores were designed asymmetrically, with a thinner palatal side. In contrast to similarly shaped metallic RDPs, all the edges are rounded, as is usual in ceramic engineering structures. For the post, the concept of a cylindrical anchor with a conical tip was adopted; this enables good retention with minimum risk of root cleavage.[6] In Fig. 1 a cylindro-conical outline for the root portion of the post system and the retentively shaped coronal portion is presented.

Due to the small size and complicated shape of RDPs, low-pressure injection moulding (LPIM) was selected as the shaping technique for the forming. LPIM is a cost-effective near-net-shaping method for producing complex-shaped ceramics, and offers several advantages over other methods of forming ceramics.[7,8] This technology involves compounding the starting powder to form high-solids-loading feedstocks, shaping of the green parts with LPIM, extraction of the binder, and sintering. All the steps should be optimised to prevent the formation of defects, which may subsequently influence the mechanical properties and the reliability of the sintered product. In addition, after sintering, RDPs can be HIPed to improve their mechanical properties.

The purpose of our work was to evaluate the effect of some processing variables, such as the powder loading of the suspensions and the molding conditions, on the fracture resistance and

* Corresponding author. Tel.: +386 1 477 48 67.
E-mail address: ales.dakskobler@ijs.si (A. Dakskobler).

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Fig. 1. Root-dental-post with diameter 1.5 mm showing retentive post head with apical, middle, and coronal ring structures.

Weibull modulus of sintered and additionally HIPed zirconia (Y-TZP) RDPs with a core.

2. Experimental work

Experimental RDPs were produced from commercially available Y-TZP powder containing 3% Y_2O_3 in the solid solution (TZ-3YSE; Tosoh Corp., Tokyo, Japan). The material also contained 0.25% alumina to suppress the tetragonal-monoclinic transformation during aging in an aqueous environment.

Two suspensions with solids loadings of 56.5 and 58.2 vol.% of zirconia powder were prepared. Their compositions are presented in Table 1. KX 1313 paraffin wax (Zschimmer & Schwarz, Germany) was used as the liquid medium and stearic acid (Kemika, Croatia) was added as a surface-active agent. The suspensions were prepared by blending zirconia powder in a molten mixture of KX1313 paraffin and stearic acid on a three-roller mill at 80 °C. These suspensions were injection molded, at a pressure ranging from 0.2 to 0.6 MPa and a temperature of 67–74 °C, into a steel mold to form RDPs. The prepared specimens were thermally debinded in a powder bath at 200 °C for 1 h. The heating rate was 0.2 °C/min. After thermal debinding the specimens were sintered at 1550 °C for 2 h. Some of the specimens were also hot isostatically pressed at 1450 °C under a pressure of 100 MPa, followed by a heat treatment at 1300 °C to reoxidize the material.

Table 1
Composition of prepared suspensions

	Powder (vol.%)	Paraffin (vol.%)	Stearic acid (vol.%)
Suspension 1 (S1)	56.5	39.7	3.8
Suspension 2 (S2)	58.2	37.1	4.7

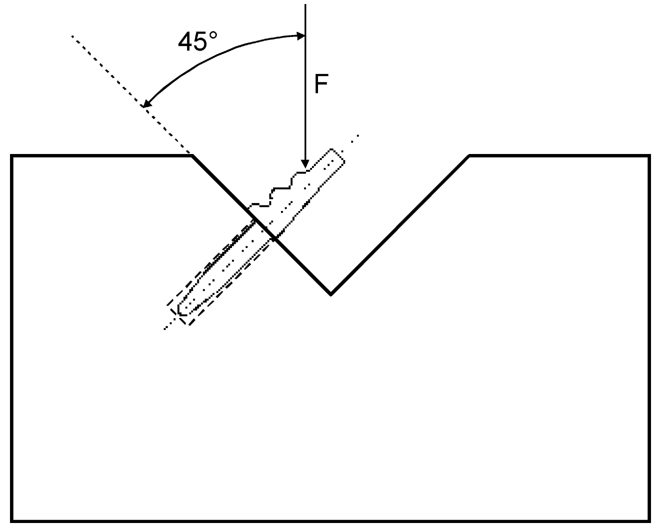

Fig. 2. Schematic presentation of mechanical testing of root-dental-posts.

The flow curves of the suspensions were analyzed with a rotational viscotester (Haake VT 500, Germany) using the cone-and-plate PK-2 measuring system. The temperature of the suspensions during the measurements was 70 °C.

Microstructural studies of the sintered specimens were performed using an optical microscope (Olympus BX60, Japan). A scanning electron microscope (Jeol 5800, Japan) was used for a microstructural characterization of the polished and thermally etched (1420 °C, 45 min) specimens.

The fracture resistance was determined by measuring the load on RDPs inserted into artificial root canals. The artificial root canals were drilled in a stainless steel mold in accordance with the previously reported method.[9] The diameter of the drilled holes corresponded to the radicular part of the post's diameter (1.5 mm). The depth of the canals was such that the post seated in the canal up to the apical retentive ring. The posts were loaded in a universal testing machine (Model 4301; Instron Corp., Canton, MA) at an inclination of 45°, with the loading tip resting on the coronal retentive ring. This is schematically presented in Fig. 2. In this way the loaded portions of the posts were of uniform length (5 mm). The posts were loaded at a constant cross-head speed of 1 mm/min. The fracture load (N) necessary to cause the failure of the post was recorded.

Weibull statistics[10] were used to determine the differences between the fracture loads of the different groups of specimens. The variability was analyzed using the two-parameter Weibull distribution function[11]:

$$P(F) = 1 - \exp\left(-\frac{F}{F_0}\right)^m$$

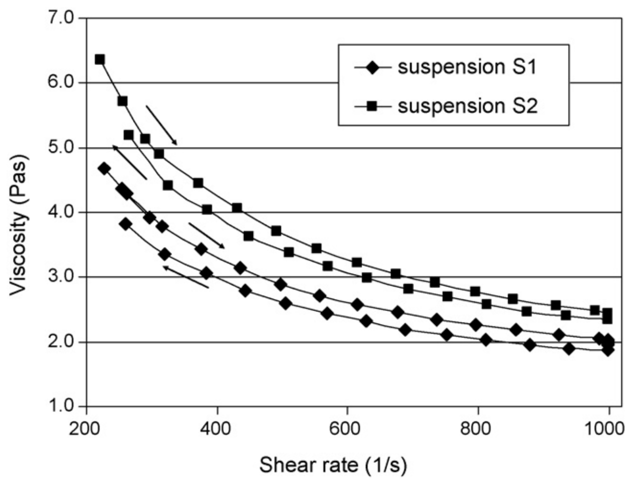

Fig. 3. Flow curves of the suspension S1 and the suspension S2 at temperature 70 °C.

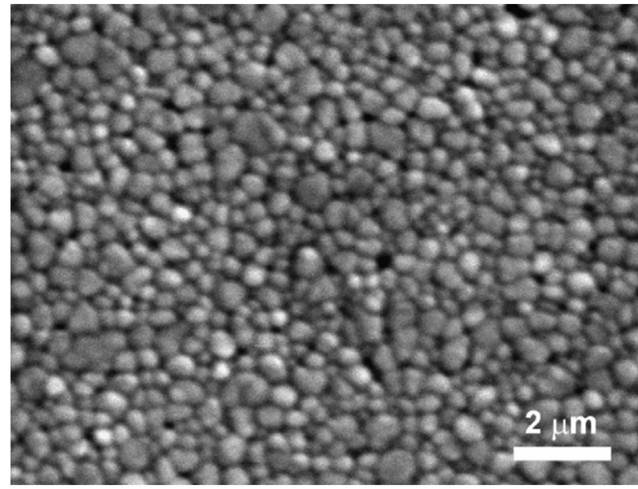

Fig. 5. Microstructures of sintered specimens S1.

where $P(F)$ is the fracture probability, F is the fracture load, F_0 is the characteristic load to fracture at a fracture probability of 63.2%, and m is the Weibull modulus, which is the slope of the $\ln(\ln(1/1-P))$ versus $\ln F$ plots. Statistical software (Weibull ++, Version 5.0; ReliaSoft Corp., USA) was used to estimate the two-parameter Weibull distribution function.

3. Results and discussion

Fig. 3 presents the up and down flow curves for the prepared suspensions S1 and S2 (for the compositions, see Table 1). It is clear from the flow curves that across the whole measured range the viscosity is higher for the suspension with the higher solids loading (suspension S2). This small difference in the solids loading between the suspensions caused a considerable difference in the suspension viscosity, because at high solids loadings the viscosity of the suspensions is very dependent on the percentage of powder in the suspension. Furthermore, both suspensions exhibited pseudo-plastic behavior (shear thinning) and a comparable time dependence (thixotropy), which is defined as the difference between the up and down flow curves.

The appropriate molding conditions for the S1 suspension were determined by mapping the defects present in the sintered specimens that were shaped at different temperatures and pressures. Fig. 3 presents three characteristic types of defects originating from the inappropriate molding conditions of the specimens. Large voids (Fig. 4a) and cracks extending perpendicular to the molding direction (Fig. 4b) were readily observed when the molding temperature was high and the injection pressure was low. In contrast, a low molding temperature at a high injection pressure resulted in large, elongated subsurface pores (Fig. 4c). By adjusting the molding temperature and the injection pressure these characteristic defects that relate to the injection-molding step were eliminated. The optimum conditions for the injection molding of this suspension were determined to be 69 °C and a pressure of 4 bar.

The microstructure of the sintered specimens prepared from a suspension containing 56.5 vol.% of zirconia powder (suspension S1) revealed a dense microstructure with small equiaxed grains (Fig. 5), a few fine pores and a porous core. This microstructure is presented in Fig. 6. The origin of the porous core is the low solids loading of the suspension. During the molding the mold is filled with a suspension, which has a higher temperature than the mold, and the suspension is cooled from the outer part of the specimen toward the interior. At lower solids loadings the suspension shrinks more, and because of the shrinkage of the paraffin a porous core is formed in the specimens.

The second set of specimens was prepared from suspension S2, which contained a larger amount of zirconia powder (58.2 vol.%). Due to the higher viscosity of this suspension, the molding conditions were slightly altered to avoid the formation

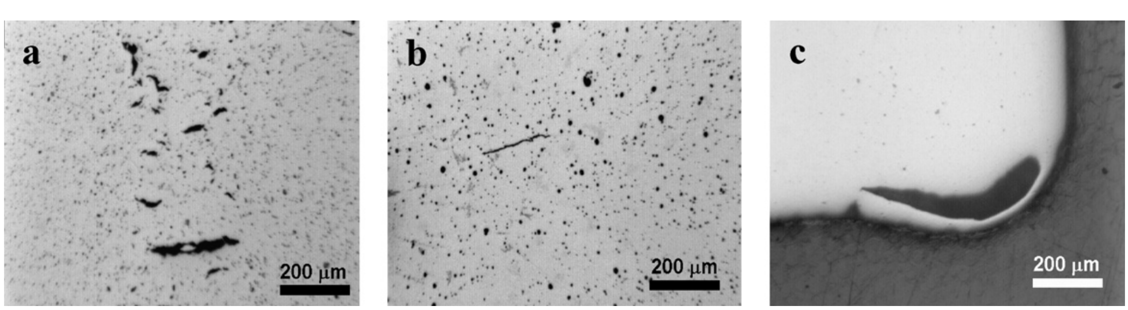

Fig. 4. Characteristic defects originating from the injection molding processing step: (a) large voids; (b)cracks and (c) large pores due to entrapped air during molding step.

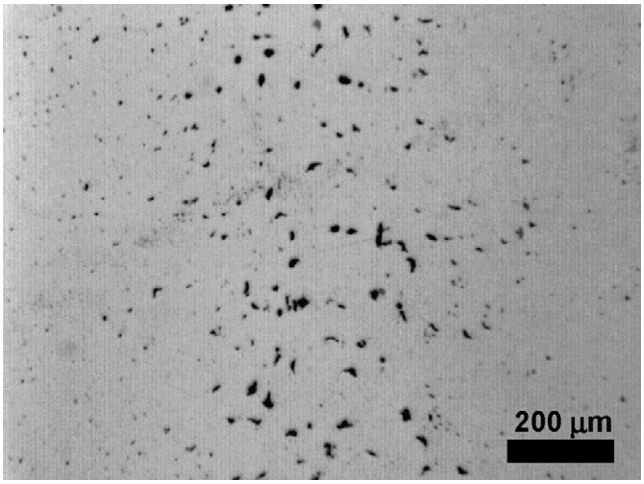

Fig. 6. Optical micrograph of porous core present in sintered specimens S1.

of large forming defects, as presented in Fig. 4. The appropriate conditions were found to be 72 °C and a pressure of 4 bar.

Fig. 7 presents an optical micrograph of a polished surface of sintered specimens prepared from suspension S2. The micrograph reveals a very dense structure, indicating that the formation of a porous core was prevented. Based on these results we can conclude that a higher solids loading of the suspension resulted in denser particle packing. The microstructural characterization revealed small equiaxed grains with a few fine pores. The sintered microstructure of the specimens prepared from suspension S2 is the same as the microstructure presented in Fig. 5, i.e., no difference in the grain size was detected.

The sintered specimens were mechanically tested to determine the effect of different solids loadings of the suspensions on the fracture resistance and the Weibull modulus. The average sintered fracture resistance and the standard deviations were 370 N (± 95 N) for the sintered specimens prepared from suspension S1 (specimens S1) and 440 N (± 82 N) for the sintered specimens prepared from suspension S2 (specimens S2). The Weibull plots for these two series of specimens are presented in Fig. 8. The Weibull modulus for the sintered S1 specimens

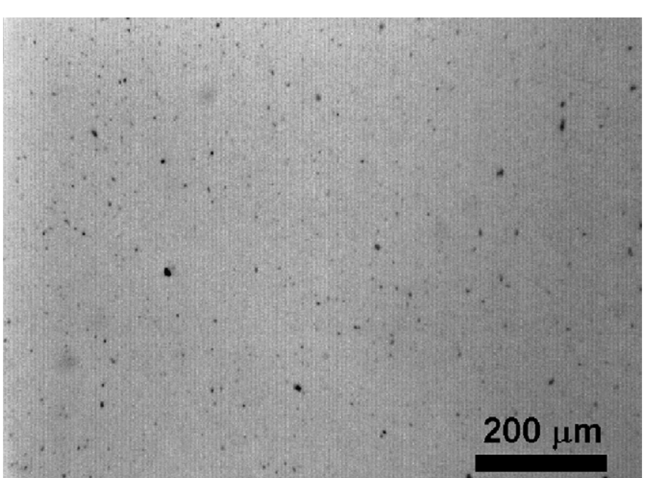

Fig. 7. Optical micrograph of sintered specimens S2.

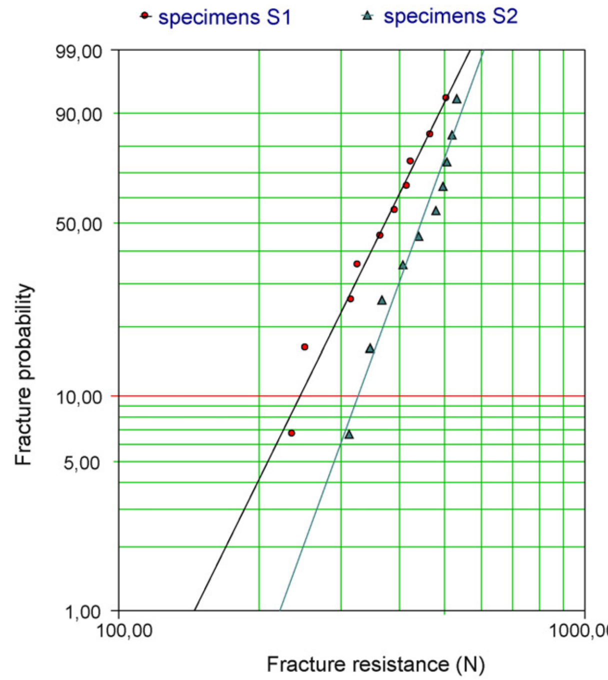

Fig. 8. Weibull plot for sintered specimens S1 and specimens S2.

was 4.50 and for the sintered S2 specimens it was 6.08. The higher strength and Weibull modulus of the S2 specimens are a result of the improved microstructure in comparison with the microstructure of the S1 specimens. Although the Weibull modulus of both series of sintered specimens was rather low, the pronounced effect of an improved particle packing due to a higher solids loading of the starting suspension can be determined and a higher modulus was measured for specimens S2.

In order to further improve the performance of the RDPs some of the sintered specimens were also HIPed, and the effect of this additional processing step on the microstructure, fracture resistance and Weibull modulus was determined. In Fig. 9a and b are the microstructures of additionally HIPed S1 and S2 specimens. Both microstructures exhibited a decreased amount of fine porosity, while the grain size remained almost the same. However, HIPing of the sintered S1 specimens did not affect their porous core, which remained the same as that presented in Fig. 6. In spite of this the HIPed S1 specimens revealed an increased average fracture resistance of 414 N (± 91 N). Also, an increased fracture resistance of 492 N (± 56 N) was measured for the S2 specimens. The Weibull plots for the additionally HIPed specimens are presented in Fig. 10. The values for the S1 and S2 specimens were 4.59 and 10.27, respectively. If we compare the Weibull distributions for the sintered and the sintered-and-HIPed S1 specimens (Figs. 8 and 10), the Weibull modulus did not change after the HIPing, only the average fracture resistance was shifted to higher values. However, we can see from Fig. 10 that two of the measured values for the S1 specimens are significantly smaller, which decreased the Weibull modulus considerably. Similarly low strengths were also measured for the S1 sintered specimens, indicating that occasionally in some S1 specimens larger defects are present, and these cannot be eliminated by additional HIPing. In the case of the S2 specimens, both

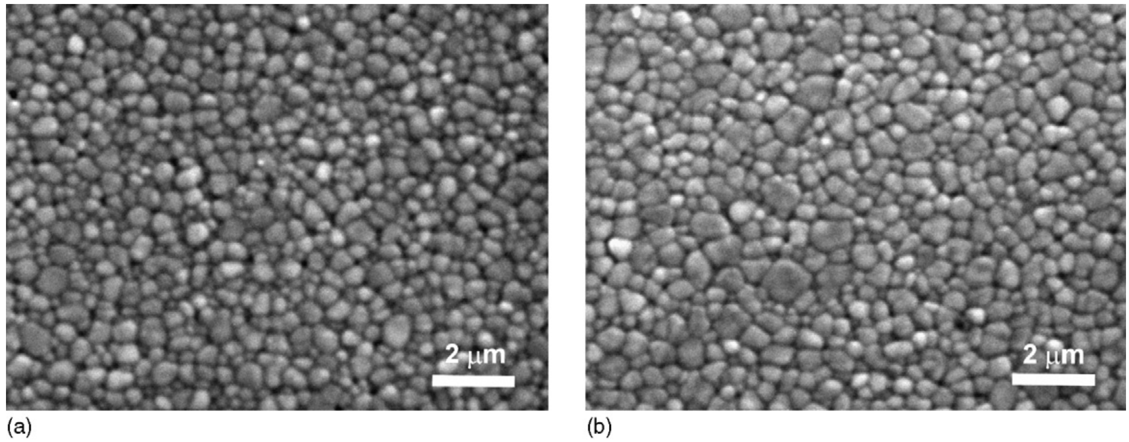

Fig. 9. Microstructures of additionally HIP specimens: (a) specimen S1 and (b) specimen S2.

parameters were increased and the measured Weibull modulus for the HIPed specimens was high. From these results it can be concluded that HIPing has an important effect on the reliability of the specimens, but just in the case when the defects in the sintered specimens are decreased to a minimum, as was the case for the S2 specimens.

Based on the low Weibull modulus one would expect larger (several tens of microns) defects in the fracture surface of sintered RDPs that exhibit low fracture loads. However, an SEM analysis of the fracture surfaces of the tested RDPs (specimens S1) revealed that in these specimens the fracture also originated from small, few-micrometer-sized surface flaws that must have been sub-critically grown before the catastrophic failure. A typical example of such a surface defect is shown in Fig. 11. This results indicate that the low Weibull modulus of the tested specimens most probably originates from the testing method used for determining the fracture load (see Section 2), which was designed in such a way as to mimic clinical conditions.

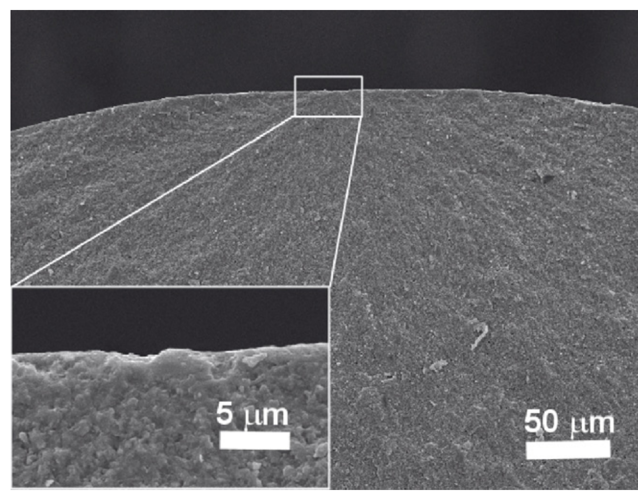

Fig. 11. Average fracture surface of sintered specimens S1.

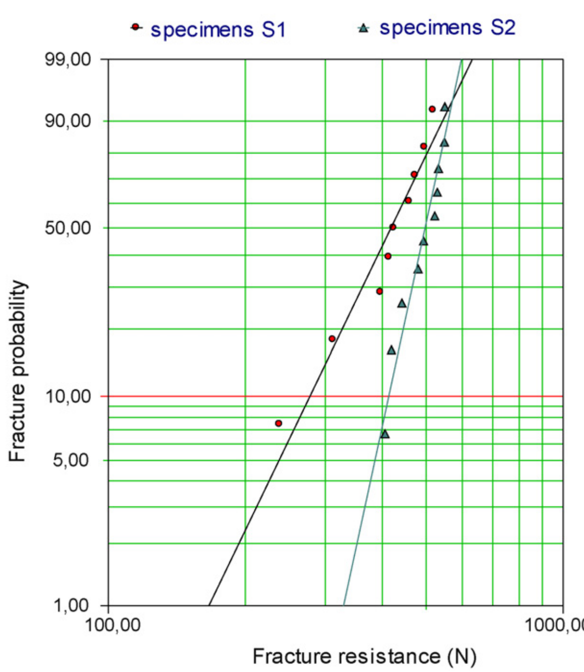

Fig. 10. Weibull plot for sintered and additionally HIPed specimens: S1 and specimens S2.

4. Conclusions

We have successfully produced zirconia root dental posts (RDPs) for affixing the crown. A low-pressure powder-injection-molding technique was used to form the green parts. The production process greatly affects the fracture resistance and the reliability of the sintered specimens. By mapping the characteristic defect origins the optimum molding conditions were determined, resulting in the elimination of large shaping defects. The solids loading of the suspension was found to be an important factor affecting the reliability and fracture resistance. The solids loading must be as high as possible to prevent the formation of a porous core, because the effect of a porous core on the reliability cannot be reduced, even with additional hot isostatic pressing (HIPing). In the case of a suspension with a very high solids loading (suspension S2), the formation of a porous core was prevented in the sintered parts due to denser particle packing. In combination with additional HIPing, this resulted in an improved fracture resistance and a large Weibull modulus.

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