Original Paper

Glass fibres from mechanically shaped preforms

Raimund Dorn, Armin Baumgärtner, Anca Gutu-Nelle, Werner Rehm, Reinhold Schneider, Sigrun Schneider and Herbert Haupt
Standard Elektrik Lorenz AG, Research Centre, Stuttgart (FRG)

A novel method for the production of SiO_2 fibres is presented, based on the MSP process (Mechanically Shaping of Preforms). This process allows the separation of powder production and preform shaping. High dispersive GeO_2-doped and undoped silica powders with a pile-up density of about 20 g/l are employed to provide step-index profiles. The predensified powder is compressed to a porous preform (70 to 90 % porosity) which is dried and consolidated in a sintering furnace and then drawn to an optical fibre. The fibres exhibit lowest attenuation of 6 dB/km at present.

Herstellung von Glasfaservorformen durch mechanische Formgebung

Es wird ein neuartiges Verfahren für die Herstellung von SiO_2-Fasern vorgestellt, das auf dem MSP-Prozeß (Mechanically Shaping of Preforms) beruht. Dieses Verfahren gestattet eine Trennung der Pulver- und der Vorformherstellung. Hochdisperse GeO_2-dotierte und undotierte SiO_2-Pulver mit einer Schüttdichte von ungefähr 20 g/l werden verwendet, um Stufenindexprofile einzustellen. Die verdichteten Pulver werden zu porösen Vorformen (70 bis 90 % Porosität) gepreßt, die nach dem Trocknen gesintert und dann zu optischen Fasern ausgezogen werden. Die Fasern weisen eine minimale Dämpfung von bisher 6 dB/km auf.

1. Introduction

In standard glass fibre production processes (MCVD = Modified Chemical Vapor Deposition, OVPO = Outside Vapor-Phase Deposition, VAD = Vapor Axial Deposition), the deposition rate is limited to a few grams per minute due to the need to optimize reaction, deposition and sintering simultaneously. The aim of the MSP (Mechanically Shaping of Preforms) process is to reduce production costs by separating powder production and preform shaping. The different production steps are shown in figures 1a to d:

– starting with powder production by flame hydrolysis,

– the highly dispersive powder is filled into a cylindrical mould,

– the predensified powder is compressed to a porous preform in an isostatic press,

– the porous preform is dried and consolidated in a sintering furnace and then drawn to an optical fibre.

2. Experiments and results

Production of the highly dispersive silica powder is performed by flame hydrolysis: $SiCl_4$ or a mixture of $SiCl_4$ and $GeCl_4$ are oxidized in the oxyhydrogen flame. The SEL (Standard Elektrik Lorenz AG) Research Centre obtains GeO_2-doped and undoped silica powders from Degussa AG, Hanau. Commercial as well as pure materials are supplied. Some metal concentrations are given in table 1. The specific surface area ranges from 50 to 350 m^2/g with a pile-up density of about 20 g/l.

An essential part of the procedure is to compact the prefabricated powder to a porous preform. Doped and undoped powders are filled simultaneously in cylindrical moulds in order to form the desired refractive index profile in the preform. The homogeneity of filling determines the final geometric control. This step increases the density from 20 up to 100 to 200 g/l. By isostatic compression an irregular shape of preform is obtained despite of homogeneous filling (figure 2, left side). Therefore, the predensified powders are pressed radially in an isostatic press by fixing the length of the mould. The result of radial pressing is presented in figure 2 on the right side. The progress achieved is obvious. Apart from the very end of the radially pressed preform the diameter fluctuation is lower than ± 1 %. If a central rod is inserted into the pressing mould at the filling step a tube can be produced (figure 3).

The mechanical stability of the preforms is sufficient for handling at further procedures. The density of the compressed material is a function of the applied pressure. By variation of pressure the density of the compacts can be controlled between 10 and 30 % relative to fused silica (figure 4).

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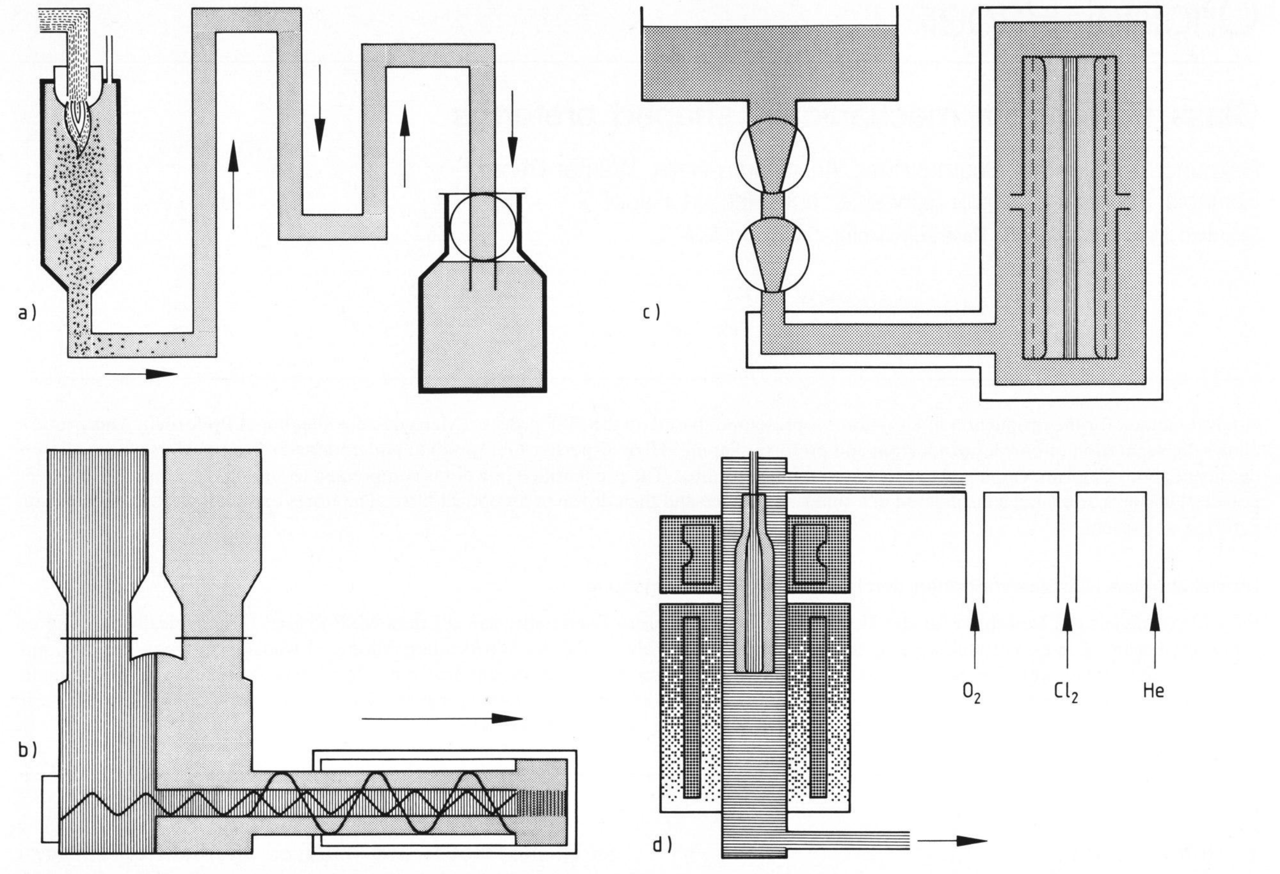

Figures 1a to d. Main processes involved in producing mechanically shaped fibre preforms; a) powder production, b) filling into the mould, c) compression, d) sintering.

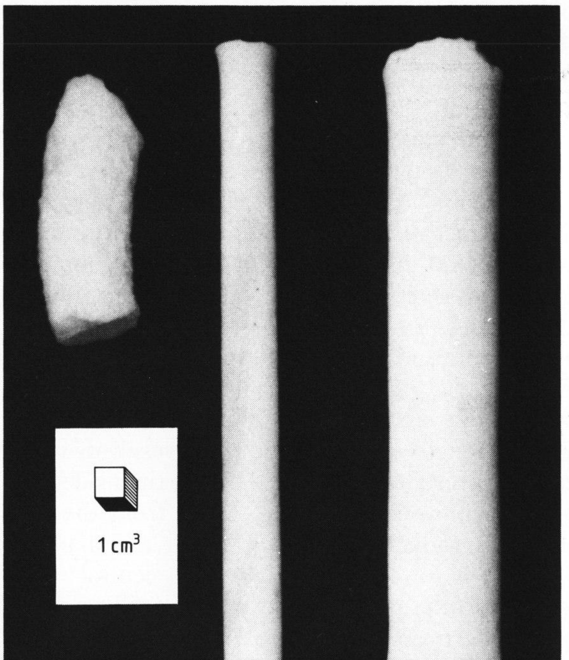

Figure 2. Compacts of silicon dioxide powders formed by isostatic (left) and radial compression (middle and right). Relative density: 20 to 25 %.

Table 1. Transition metal impurities in silica

concentration in ppb		
metal	Aerosil	pure material
Fe	2000	10 to 20
Ni	500	< 3
Cr	?	< 2
Co	40	< 2
V	100	< 5
Pb	?	5 to 10

The porosity of 70 to 90 % allows efficient drying and purifying of the soot. In order to analyse the microstructure SEM (Scanning Electron Microscope) micrographs of the pressed material are performed (for an example see figure 5). The secondary particles observed are spherical in shape with a mean diameter of 250 nm. This is approximately the same size as reported for VAD-material [1].

Since the powders are produced by flame hydrolysis and for a considerable time are in contact with an atmosphere containing water vapour they have a water/hydroxyl content of 1 to 3 % after compacting. The water is removed by annealing in an oxygen atmosphere at temperatures up to 800 °C,

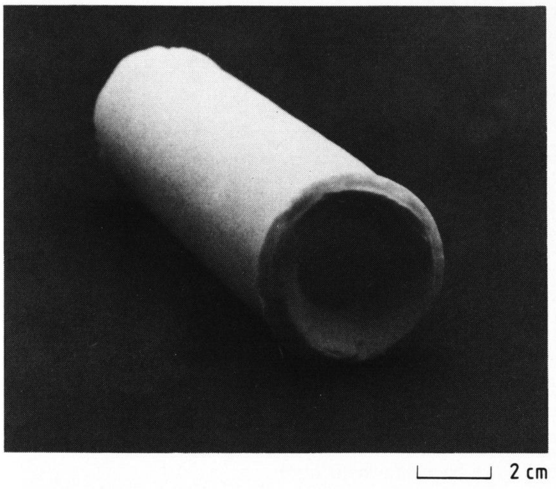

Figure 3. Porous tube made by radial compression.

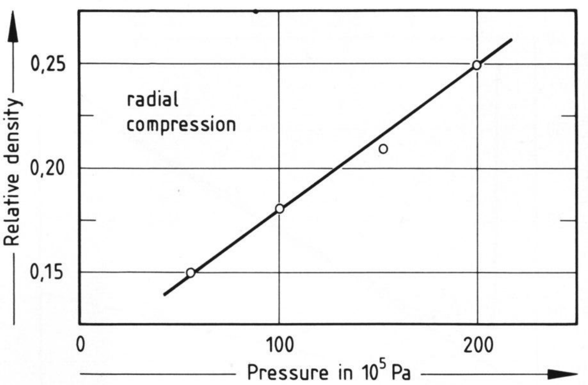

Figure 4. Density of oxide powder compacts as a function of radial pressure.

followed by chlorination at higher temperatures. Chlorination also removes remaining metal oxides which react to volatile chlorides. After this physical and chemical treatment, the porous preform is consolidated at temperatures between 1300 and 1700 °C depending on material composition.

An elementary model describes the sintering process as viscous flow driven by surface energy reduction [2]. The final stage of the sintering is the collapsing process of the closed pores embedded in the glass material. Whether the closed pores collapse or not depends on the environmental gas atmosphere. Helium is the most favourable sintering atmosphere as it has the largest critical bubble diameter of about 0.5 μm [3].

Figure 6 presents different consolidated preforms with the neck-down regions from fibre drawing and illustrates the improvement in reducing the number of bubbles. The bubble content can be characterised by detecting the light scattered orthogonal to a laser beam passing along the axis of the preform. The progress achieved by optimizing drying and sintering parameters is demonstrated in figure 7.

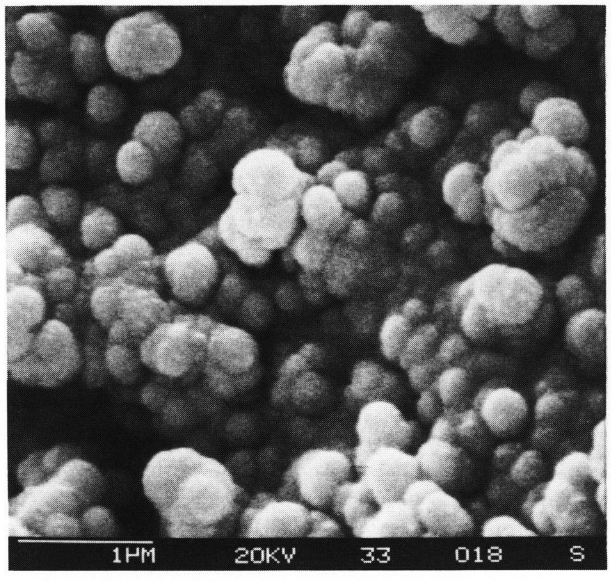

Figure 5. SEM micrograph of porous preform.

Finally the consolidated preform is drawn to a fibre with a diameter of 125 μm. The glass fibre is coated with silicone. For characterization of the fibres the spectral attenuation is measured. Figure 8 shows the spectral attenuation of one typical so-called PCS fibre (plastic clad silica). The optical attenuation is displayed versus $1/\lambda^4$ in order to separate the different absorption mechanisms. Rayleigh scattering is supposed to be the lower limit of the optical attenuation of every glass fibre and is given by

$$A_R = A_{R0} \cdot \lambda^{-4}$$

where λ = wavelength.

A plot A vs λ^{-4}, however, displays pure Rayleigh scattering as a straight line with its slope defining A_{R0}. A complete description of the attenuation is

$$A = A_R + A\,\lambda + A_c$$

where $A\,\lambda$ = any wavelength dependent impurity or intrinsic absorption and A_c = any constant absorption.

Therefore, any different wavelength dependent absorption mechanism can be detected by deviations from the straight line.

From figure 8 one can extract a lowest absorption of 6 dB/km with a wavelength independent attenuation of about 2 dB/km. The steep increase of attenuation at $\lambda > 850$ nm is caused by residual water in silica. The residual OH content may be caused by the humidity of the gas in the sintering step. Studies have shown that 0.5 ppm of water in the gas stream would result in about 1 ppm OH in the fibre [4]. Consequently, it should be possible to reduce the OH content further by drying the helium carefully.

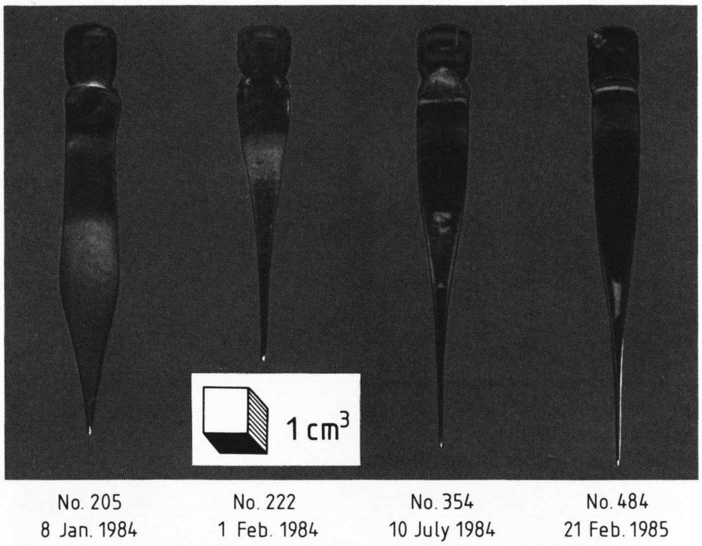

Figure 6. Undoped and Ge-doped MSP preforms that have been drawn to fibres.

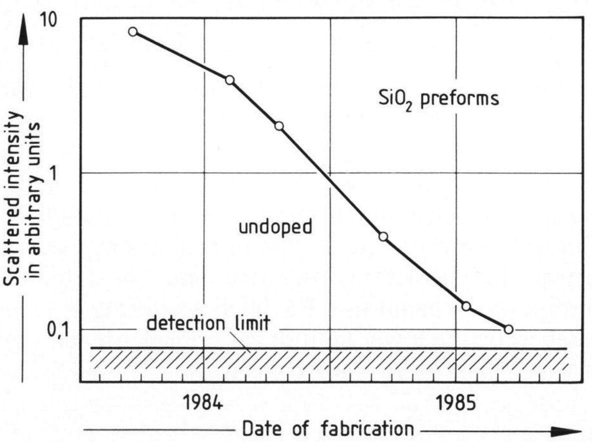

Figure 7. Tendency of light scattering at bubbles in MSP preforms.

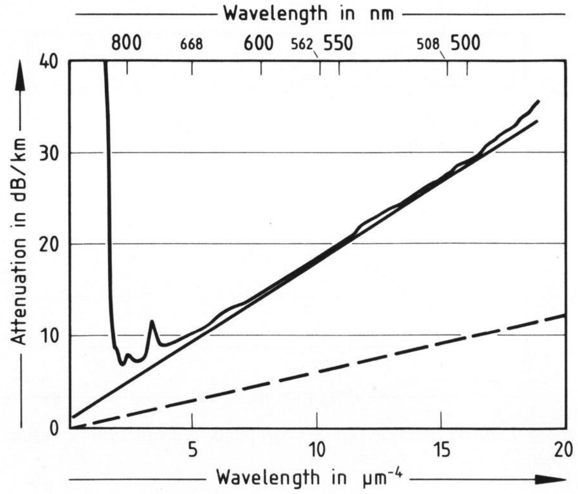

Figure 8. Spectral attenuation of PCS fibre prepared by MSP process.

3. Conclusions

The MSP process can, using cold radial compression, produce preforms with a porosity comparable to VAD or OVPO-material. Diameter control (± 1 %) is satisfactory. Preform size will have to be increased from the present 180 g to about 2.5 kg, corresponding to a fibre length of 100 km. Further improvements in the purity of the powder production as well as in powder operating in the following procedures will reduce fibre attenuation and will provide the basis for the production of high quality MSP fibres.

4. References

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