Contributed paper

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Tensile strength of tailored optical fibres

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Data are presented characterising a few groups of compound glass, special, optical fibres designed for photonic functional components and sensors. Tensile strengths exceeding 2 GPa were obtained in these fibres by careful multi-parameter optimisation of the modified multi-crucible technological process. The dependence of tensile strength on fibre material and particular process parameters were measured and assessed for a broad range of process modifications, giving several basic answers concerning the phenomena underpinning the building of internal strength of an optical fibre. Not only the resulting tensile strength was treated as a measure of technological quality but also as an integrated indicator of building a fibre of engineered characteristics from quite distant materials. The difference of thermal expansion coefficients as large as 20%, for some core-cladding glass sets of adjusted technological lengths, resulted in quite durable samples. Multi-layered and shaped-noncircular-core optical fibres showed the strength lesser by 10–40% to reference ones. The expected lifetime characteristics of a reference multicomponent glass fibre, after prooftesting with the load 0.35 GPa was estimated for 25 years under the service load of 0.1 GPa. Analogous measured and calculated data for high silica fibre give service load around 0.2 GPa or only two times bigger than for the multicomponent glass fibre case. The domination of high silica fibres is revealed at higher loads.

Keywords: compound and doped glasses, optical fibres, multi-crucible technology, tensile strength, fibre technology.

1. Introduction, origins of fibre fracture

A tailored optical fibre [1–3] is the one made of compound (soft) glass which may have complex internal structure in terms of geometry (mechanical), refractive properties (optical) and other (physical, chemical). The simplest solutions to tailored optical fibres may resemble the ones used for telecommunication purposes, but with different guidance properties. The most complex ones may have extremely complicated refractive index profiles, possess eccentrically placed cores, have strictly designed modal spectrum or be multi-core [4,5]. Prospective applications of such fibres are purely photonic sensors. We are considering multicomponent glass, optical fibres, manufactured during a modified multicrucible process (referred shortly to as the MMC) [4]. The strength of drawn optical fibres depends on several technological and environmental parameters. The major ones, according to our own experience [2,3] and reference data [6–9], are: glass quality, parameters convergence of core and cladding, furnace quality, technological parameters, fibre complexity, mechanical damage, type of coating material, covering quality and thickness of primary jacket.

The parameters influencing the tensile strength of optical fibres are so numerable and their impact accidental, thus one can estimate the strength only with a certain probability. The mechanically weak points, which can be found in any fibre, are due to the inhomogeneity of the oxides in the glass, perturbations on the fibre surface during the pulling process, dust particles, OH particles, devitrification micro-centres, etc. Most of these irregularities can be accounted for in a statistical manner. The fibre can be fractured more easily at some of these irregularities, than anywhere else. Thus, the irregularities decide usually on the strength. Removing most of the irregularities, with the most critical ones located on the fibre surface, leads to the natural tensile strength of the perfect flawless glass filament.

The network forming cations such as Si^{4+}, Al^{3+}, B^{3+} that tend to form tetrahedra or other coordinated networks with oxygen usually strengthen the glass when added to the compound system. Other ions, network modifiers (like alkali ions), which lie in open space, in the network, usually weaken the glass, when added in excessive amounts. The ultimate strength of glass is determined by the bonding forces within its material structure. The variety and complexity of glass microstructure preclude accurate determination of total strength by calculating the aggregate of bonding forces, especially in compound glasses. However, the analysis of dissociation energies of the bonds and of molecular packing densities, leads to theoretically calculated, approximate values of Young's modulus for simpler glasses [8,9]. Till now, the ultimate strength of glass is only of theoretical interest. Here, we investigated practical

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strength of glass filaments, when these filaments were subject to stress, and failure occurred. The failure occurred when the ultimate strength limit was exceeded at some length of the structure. The failure mechanism depends on the stress distribution and, thus, on the micro-heterogeneity of the glass. Micro-heterogeneity is, in turn, a direct consequence of local phase separations in glass, glass composition, thermal history, melt atmosphere, level of impurities, presence of water, etc. The stress concentrations occur at the micro-nonhomogeneities with the domination on external surfaces, where there are micro-flaws caused by the natural process of surface formation.

Exposing the glass surface to the environment and handling it, is a cause for a large number of surface flaws to develop. The sizes of the flaws vary to some extent statistically, what show the measurements on uncoated optical fibres. Surface protection diminishes the flaw dimensions considerably (of an order of magnitude). The fracture strength of a particular length of glass filament depends on the dominant flaw, the biggest one, where there is the biggest concentration of stress. When mechanical flaws are eliminated (through very careful handling of the surface), the remaining flaws are due to chemical and structural causes. These are smaller than mechanical ones. They can be partially eliminated through the precise glass homogenisation.

The major aim of the paper is to assess some basic rules underlying the statistical nature of tensile strength of compound glass optical fibres of simple as well as of more complex internal structure. For the first time, the full mechanical data on tailored MMC optical fibres are made available. The paper covers a number of metrological and technological problems concerning the interdependencies between technological parameters of fibre manufacturing process, fibre structure and material, and the tensile strength of resulting fibre. The following technological parameters were investigated: atmosphere quality in the furnace, glass quality, outflow meniscus shape, discrepancy of glass parameters for core and cladding etc. The tensile strength of the following multicomponent glass optical fibres was measured: multi-layer, circular, triangular and square core ones. Predictions were presented concerning the expected lifetime of the best measured special optical fibres. These results were compared with analogous ones for high silica fibres.

2. Physical background and technical description of tensile strength of optical fibre

Glass is a strong, elastic material, which obeys Hooke's Law $\sigma = E\varepsilon$, where σ is the stress (tension or compression), E is the Young's modulus, $\varepsilon = \Delta l/l$ is the strain. It is also very brittle. The ultimate strength is determined by the bonding forces of the constituent atoms. The theoretical cohesive strength (here failure stress) can be expressed by Griffith equation: $\sigma^2 = 2\gamma E/\pi a$ [10], where γ is the surface (activation) energy of glass, a is the atomic spacing or bond distance. The ultimate strength of silica glass depends on the Si–O bond, with the distance 0.16 nm [8], and thus is over 18 GPa. Practical strength depends on the surface quality. It is assumed that the flaws, according to the classical Griffith description, are narrow cracks, with small radii of curvature at their tips, where applied stresses are concentrated, through the leverage effect. Under certain circumstances (presence of threshold stress and water) the flaws will propagate. If the flaws are fine, the elasticity theory applies, and the breaking stress at the flaw is described with the same Griffith equation as the ultimate strength but a denotes flaw length [6]. To arrive at this equation it is assumed that the cracks are fine, straight with an elliptical cross section. The stress S is applied perpendicularly to the crack or along to the fibre axis. The stress at the crack tip in the direction of applied stress S is $\sigma = S(1 + 2a/w)$, where a is the crack length, w is the half crack width. Radius of curvature at the crack tip is $r = w^2/a$, with $a >> w$, thus

$$\sigma = 2S(a/r)^{1/2} = (2\gamma E/\pi a)^{1/2} = ca^{-1/2} \qquad (1)$$

where c = const. It is assumed that r, which is of atomic dimensions, does not depend on a, and that the fracture stress S_f is inversely proportional to the square root of a. Let us assume that the constant c depends mainly on the Young's modulus, because the glass surface energies differ only slightly among optical fibres of similar quality, prepared by similar methods. These constant values, calculated from glass data [9] are: c = 7–7.4 for high silica fibres, c = 6.2–7 for SLS, SBS and light compound glass fibres, c = 5.7–6 for fibres made of the lowest E value glass (E = 45–50 GPa), c = 7.6–8.5 for the highest E value glasses (E = 80–100 GPa). Exemplary glasses of the high E values and suitable for some speciality optical fibres are light as well as heavy barium crowns, for example BK and BaCK. Exemplary glasses of the low E value and suitable for some speciality optical fibres are very light fluoride crowns, with the density below $\rho = 2.3\times10^3$ kg/m^3, for example FLK. Most flint glasses, suitable for speciality optical fibres, have intermediate values of Young's modulus.

The dependence of optical fibre strength versus random crack size was calculated, for a few different families of optical fibres, and presented in Fig. 1. Some of these fibres, of quoted material data, are investigated experimentally. The critical crack length is of the order of a = 2 nm for high-quality, doped silica fibres, with the tensile strength in the range of 5 GPa. The break-initiating crack has dimension of the order of a = 10 nm, for compound glass optical fibres, with average maximum tensile strength around σ = 2 GPa. The largest cracks have dimensions of the order of a = 3 nm, in the best compound glass fibres, with tensile strength in the region of σ = 3.5 GPa. The equivalent data for analogous high silica fibres is around a = 4.5 nm. The span of critical crack sizes responsible for breakage of different optical fibres of the same construction is over half

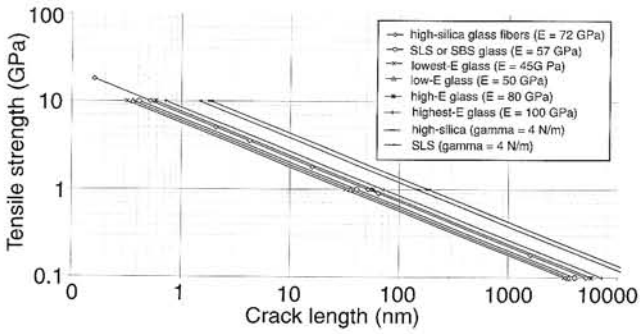

Fig. 1. Calculated tensile strength σ(GPa) of optical fibres of compound glasses as a function of the largest breaking crack length a (nm) present statistically in the glass filament, according to Griffith theory $\sigma = (2\gamma E/\pi a)^{1/2}$. Data for doped silica fibres quoted for comparison and reference. The assumption is that the surface energies are nearly equal among these glasses and are approximately $\gamma = 1.125$ (N/m). Two groups (high silica and SLS) presented for activation energies $\gamma = 4$ (N/m).

a decade, for constant strength value. For example, fibres of $\sigma = 3$ GPa strength, break due to cracks from a = 3.2 nm to a = 8 nm, or of the dimensions ratio 2.5.

Apart from glass properties, the fibre strength depends essentially on the state and quality of its surface, which is represented in Griffith equation by the surface energy. This was shown by two additional characteristics in Fig. 1, where the surface energy was assumed to be $\gamma = 4$ (N/m), against the previous value which was around unity.

The mechanical strength of an optical fibre is a time dependent parameter. It is described, in the best way, by the Weibull distribution [11], from technical point of view. It is used as an international standard. This method shows the probability of the fracture F in an optical fibre dependent on the length l, the mechanical break stress σ and the time t

$$F(l,\sigma,t) = 1 - \exp\{(l/l_o)(\sigma/\sigma_o)^a (t/t_o)^b\}, \qquad (2)$$

where a, b are the distribution constants, l_o, σ_o, t_o are the test (or nominal) constants, all of them determined experimentally. For $l = l_o$, $\sigma = \sigma_o$, $t = t_o$ $f(l_o, \sigma_o, t_o) = 1 - 1/e = 0.632$ which is the nominal value of the distribution. Typical values for the distribution constants, for high silica optical fibres, are $a = 3 \pm 1$ and $b = 0.2 \pm 0.05$, with $l_o = 20$ m, $\sigma_o = 2$ GPa, and $t_o = 1$ s [12]. Weibull distribution is usually shown without time dependence, this is for $t = t_o$. σ is plotted in logarithmic scale and probability in either linear or logarithmic (%) or $\log\{\ln[1/(1 - f(l,\sigma))]\}$, possibly $\ln\{\ln[1/(1 - f)]\}$ scales. The slope of the straight line, drawn through the measured points, gives the distribution constant a. The plot in double logarithmic scale is linear and changes from –7 to +2 (with zero for the nominal value of distribution), for the change from 0.1 to 100 on cumulative probability scale, expressed in (%).

3. Measurements of tensile strength of compound glass, tailored, optical fibres

There are not numerable measurement data available, concerning the tensile strength of compound glass optical fibres [13]. On the other hand, a lot of data is available on high silica communication fibres [9], which is due to understandable reason of overwhelming usage domination. There is no data at all, of this kind, concerning tailored optical fibres [13]. The strength of a glass filament varies statistically along its length due to the random nature of flaw depth and spatial distribution. The measurement of fibre strength is complicated by this randomisation. It requires appropriate, usually redundant, number of data to be gathered. One is trying to predict the tensile strength of a long length fibre based on measurements of confined gauge length. The measurements should cover the statistics of all kinds of flaws, even large but rare. Only in such a case, the extrapolation from one gauge length to another is valid with known accuracy. More accurate approximation of data is available if the exact nature of rare flaws is known. Since it is a destroying measurement, a test facility and simple procedures are desired not to waste more fibre than necessary. Redundant data obtained from tests are to be reduced without distorting the flaws statistics. There are three standardised measurements of fibre mechanical strength: dynamic fatigue tests, static fatigue tests and proof tests. A fatigue is strength degradation caused by stress induced corrosion. The strength specifications of a fibre include tensile strength distribution with its width, median and extreme values, time to failure versus stress level, minimum expected lifetime under service load and are obtained by processing the measurement data using Weibull statistics. The minimum lifetime is specified for particular working conditions of the fibre, including temperature, humidity, pH, etc.

Dynamic fatigue test was used to estimate the minimum lifetime of a fibre under load. A constant rate of loading was applied on the stretching machine, until fracture occurred. The loading rate was shown to affect the average strength and strength distribution in a real fibre due to fatigue effect. The used range of loading rate was from 0.1 MPa/s to 0.1 GPa/s. The fracture load was recorded as well as the diameter of the broken end of fibre. The highest quality fibres had very narrow spread of Weibull data distribution. The responsible flaws were shown to be unimodal with narrowly confined dimensions.

Static fatigue test was used on accidental samples of fibres to measure time aspect of Weibull distribution. Time to failure under constant stress was measured [7]. It has been found that the fibre fatigues, under the static loads seen in service, less than 50 MPa, by subcritical crack growth. It showed, indirectly, the crack velocity in the fibre as a function of applied force what was a measure of fibre survivability (maximum lifetime) in particular stress and moisture environment. Axial loading, analogous to the

stretching machine method, but with constant loading (rate zero) was used throughout this work.

Proof test was applied to the entire length of fully coated optical fibre on duty. The fibre from the draw machine or payoff reel was fed through the high-tension region to the low-tension take up. A system of rollers was used to exert a stress in the range of 0.2–1 GPa on the fibre as it was running through them. The test was used to truncate the distribution of flaws, since the largest flaws govern the long-length strength. It was also used to check the validity of long length strength predictions using short length testing method. The accuracy of these predictions is better when strength distribution is unimodal and parameters of Weibull distribution are determined correctly and accurately. The minimum guaranteed fibre strength, checked by applying proof stress, affects also the lifetime. Higher proof stress assures the minimum guaranteed lifetime of the proof tested fibre. Although the lifetime is greatly increased by raising the proof stress, this increases fibre breakage. The proof stress must be selected to yield both an adequate lifetime and an acceptable fibre yield. It was confirmed that the proof stress for optical fibres should be three times the service stress [12].

Figure 2 presents measurement results of tensile strength of compound glass, optical fibres manufactured by the MMC technology at "Biaglass" Co. The fibres were selected, from several batches, to be of the highest possible quality available by the technology. The process parameters and the quality of input materials were chosen to obtain the maximum tensile strength. The fibres were made to be similar to the standard multimode transmission ones. The presented plots were chosen out of a bigger series of measurements to be statistically representative for this family of fibres. The standard deviation, from the presented measurements, was approximately ±10%. The experiment data are listed below. Nearly the same data were chosen for the next experiments throughout this work, thus only the changes from this list were mentioned, where appropriate: redundant number of measured samples 250, minimum required number of measured samples was around 25, or an order of magnitude less (for the measurement to be statistically valid), the measured fibre sample length l_f = 0.5 m, the uncoated fibre diameter d_{fu} = 125 μm, the coated fibre diameter d_{fc} = 150 μm, two layers of thermally cured acrylic lacquer applied as immediate coating, silicone rubber applied as outside coating, outside diameter of the fibre d_{fo} = 1 mm, proportions of cladding/core in the fibre 125/62.5 μm, surface of glass fibre cross-section A = 12.3×10^{-6} m^2, numerical aperture in the range NA_{min} = 0.25, NA_{max} = 0.3, the quasi step index refractive profile $\alpha \approx \infty$, difference in linear expansion coefficients of core and the cladding glasses $\Delta\alpha_l$ = 3%, rate of the applied stress s_r = 35 MPa/s, which was approximately equivalent to 10 mm/min, the Young's modulus of fibre materials E_b = 55 GPa (SBS) and E_l = 57 GPa (SLS), the Poisson number ν = 0.245 for both SLS and SBS fibres, strain at break (for best samples) 3%, the ambient temperature

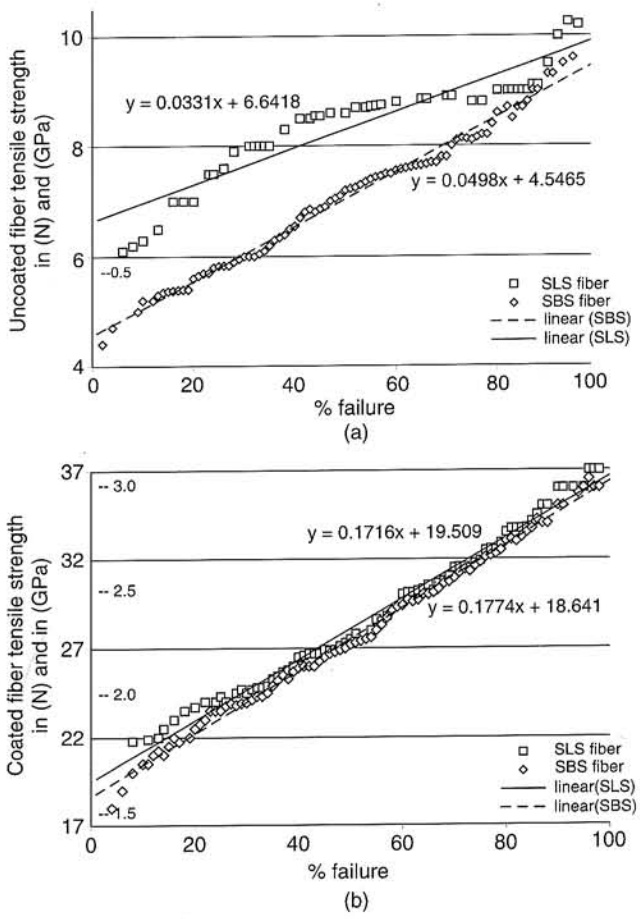

Fig. 2. Measured Weibull distributions of tensile strengths for (a) uncoated and (b) coated SLS and SBS, optical fibres of the highest quality, manufactured during the MMC process. Data presented in double linear coordinates. Experimental data discussed in the text.

20°C, the relative humidity during measurements and fibre storage after drawing h_r = 55%. The double, linear coordinate system was chosen to present the data, instead of the double logarithmic one.

The results of this experiment can be summarised as follows. The most accurate coating increases the tensile strength of the SLS and SBS fibre around four times. However, in some cases, it was around six times. The tensile strength varied, on the average, in the range of 1.5–3 GPa, for coated fibres, with the mean value of 2.2 GPa, and standard deviation around 0.1 GPa. The best fibres withstood over 3.5 kg tension, which is equivalent approximately to 300 kg/mm^2. The plots were presented in linear coordinates because the compound glass fibres are considerably weaker than high-silica ones and have more nonuniformities. Some of these unevennesses may be dampened by the logarithmic scale. Linear approximations to the measurement points (presented in the figure) can be recalculated for Weibull distribution parameters. The slope of linear approximations between uncoated and coated fibres differs approximately by a factor of four. The critical (i.e., break responsible) flaw spectrum spans the following approximate range of dimensions, for both cases: 0.1–1.5 μm and

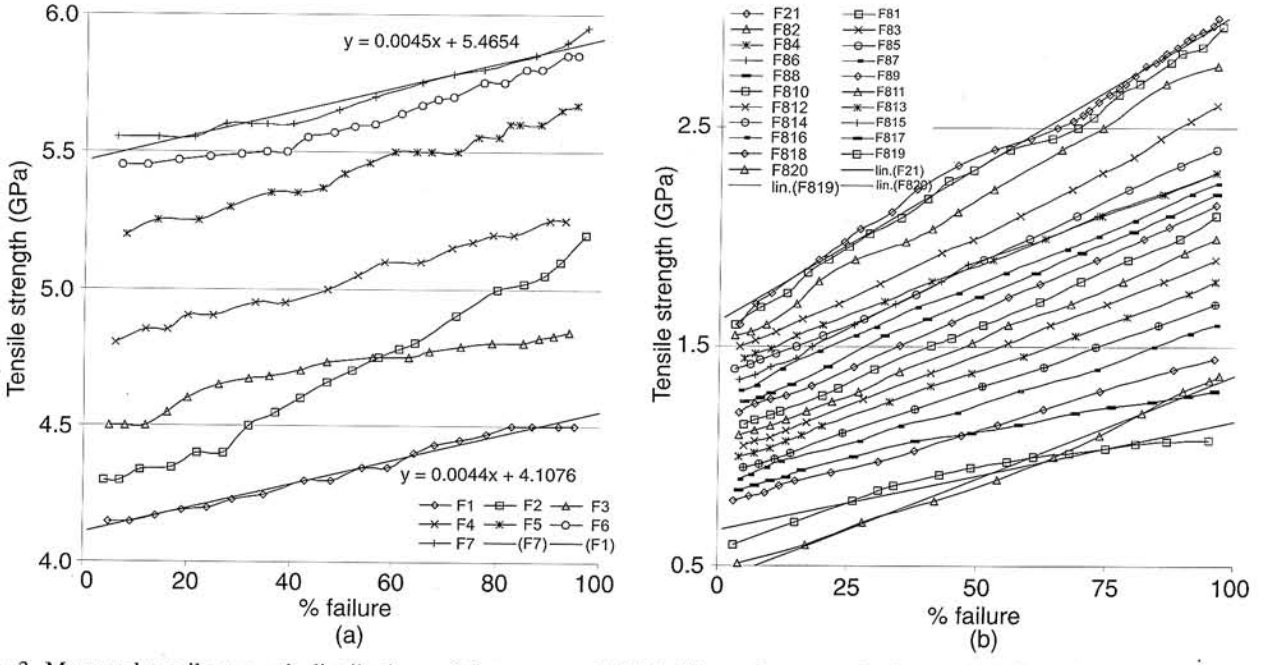

Fig. 3. Measured tensile strength distributions of factory coated, high silica, telecommunication, optical fibres from several leading manufacturers (a). Measurements performed on the same apparatus, and in the same conditions, for comparison with tailored optical fibres made of several, different, compound, glass pairs (b). The glasses are listed in Table 1.

4–40 nm. The flaw dimensions spectrum spans over one order of magnitude for coated fibres while one and a half for the uncoated, with absolute shift of two to three orders of magnitude. The SLS glass fibres seem to be slightly stronger, on the average, than the SBS glass ones, for the particular measured compositions. Choosing other compositions, slightly departing from the basic ones, however, does not support this thesis, and even tends to support the opposite. Statistical dispersion of the measurement results, for the uncoated optical fibres, was much bigger, than for the coated ones. This dispersion differed relatively by one order of magnitude between both cases. This may suggest crack building, in uncoated fibres, well after the pulling process.

The natural dimensions of flaws, built during the pulling process, seem to be of the order of a few to ten nanometers for compound glass, optical fibres. They rarely exceed one nanometer in the best, and the strongest, contemporary, high silica optical fibres, as it can be seen from Fig. 1, depicting the function $\sigma = ca^{-1/2}$ (flaw spectrum covers the region of 1.5–5 nm). The smallest flaws increase their dimensions 25 times while the largest nearly forty times in uncoated compound glass, optical fibres. This process goes on without exerting any excess stress on the uncoated fibre kept in standard ambient conditions of 20°C and relative humidity 55%, only a few hours after the drawing process.

Figure 3 shows a direct comparison between the measured results of tensile strength for doped silica fibres manufactured by CVD and compound glass ones manufactured by MMC. The measurements were performed on the same laboratory set-up to make the comparison more reliable. Measurement experiment data for both groups of the fibres were the same: sample length 0.5 m, number of measured samples in each series 50, outside diameter of fibres 125 μm, fibres coated by manufacturers (high quality coat), other data analogous to the previous cases. The data for high silica fibres were: Young's modulus 72.5 GPa, shear modulus 30 GPa, fibre material SiO_2(B,Ge,F,P), Poisson's ratio 0.21, strain at break 4%, diameter decrease at break 0.85%, the surface energy for high-silica glass $\gamma = 1.1264$ N/m. The data for compound glass fibres were: fibre proportions 125/62.5 μm, double precision coated, fibre material data gathered in Table 1. The glass compositions for tailored optical fibres include mainly the following ion systems: Na-B-Si (SBS), K-B-Si (KBS), Ge-B-Si (GeBS), Na-Ca-Si (SLS), Na-Pb-Si (SLeS), and Na-Ba-Si (SBaS). According to classical glass customary names the key to abbreviations is: BK-boron crowns, FK-fluoride crowns, PK-phosphate crowns, ZK-zinc crowns, BaLF-barium light flints, etc. Other glasses were specially synthesised for the purpose of fibre manufacturing. Crowns are multicomponent glasses with $n_d > 1.60$, $\nu_d > 50$ or $n_d < 1.60$, $\nu_d > 55$, and the others are flints. The major glass pairs to form a fibre were: SLS1-SLS2, SLBS1-SLS2, SLeS1-SBS1. Advantages of the latter pair is: considerable difference in n_d values, low temperature of technological process, very good chemical stability and resistance, exceptional mechanical qualities, very low tendency to devitrification, neutral (flat) spectral characteristic in the visible and near infrared. Other pairs combined for core and cladding of a fibre were: B4-B2, B4-BLF2-S6, B14-BK17, BF8-S1, BLF1-BK7, BLF2-BK7, BLF2-S6, BLF2-S8, F2-S6, S3-S1, S3-S2, S7-S6, S7-S8, SF5-LF7,

Table 1. Chosen parameters of proprietary multicomponent glasses used for manufacturing of MMC optical fibres at Biaglass Co.

Component Glass	SiO_2 (%w)	Al_2O_3	B_2O_3	Na_2O	K_2O	PbO	BaO	CaO	GeO_2	LiO_2	MgO	ZrO_2	ZnO	As_2O_3	Sb_2O_3	CeO_2	n_d	ν_d	α (10^{-7}/°C)	T_g (°C)	T_s (°C)	ρ (g/cm^3)	E/G (GPa)
Pure SiO_2	100																1.457	63	4.0–5.5	1200	1700	2.48	72.5/30
B2	58.8		3.5	3.5	9.9		19.0						4.7	0.6			1.540	60	80	560	725	2.9	75/31
B3	55		5.0	5.0	10.0	2.0	20.0										1.557	58	82	580	730	3.0	72/29
B4	49.7		6.0	4.6	5.0	2.1	19.9						11.4	0.8	0.5		1.569	56	72	602	730	3.0	72/29
B104	49.2		6.0	4.6	5.0	2.6	19.0						11.4	0.5	0.3	0.5	1.569	56	72	602	730	3.0	72/29
BF7	40.0	1.6	6.0	0.4	3.0	12.0	28.0										1.606	44	82	620	690	3.5	66/27
BF8	40.0	1.6	2.0	0.4	3.0	15.0	29.0						8.0	0.5	0.5		1.624	58	70	589	680	3.6	70/28
BLF1	52.8	0.8		2.2	11.0	10.3	14.1						8.0	0.5	0.3		1.563	60	71	570	700	3.2	68/28
BLF2	56.8	0.8		2.5	10.5	6.3	14.0						8.1	0.5	0.5		1.548	63	81	530	690	3.0	68/29
BLF21	60.0	0.8		2.0	14.0	10.0	11.0										1.547	63	70	550	630	2.9	70/30
BK1	60.0		20.0	11.0	6.0		2.4										1.510	64	77	547	600	2.5	74/31
BK107	68.1		11.0	11.0	6.0		2.4							1.0		0.5	1.518	64	77	560	620	2.5	78/32
BK7	66.0		13.0	11.0	6.0		2.4										1.517	64	71	559	620	2.5	78/32
F1	47.0			5.0	10.0												1.610	37	82	515	560	3.5	58/24
F2	45.6			2.6	6.0	45.3								0.3	0.2		1.620	36	82	525	570	3.6	60/25
FK3	60.0			10.0	10.0	20.0											1.465	66	82	462	530	2.3	46/18
K3	69.0		2.0	8.0	11.0	2.0							6.5	1.0			1.518	59	83	521	590	2.6	71/29
K4	69.0		2.0	6.0	10.0	5.0							6.5	1.0			1.522	60	90	550	620	2.6	71/29
K5	39.6	4.1	15.0				40.3							0.5	0.5		1.589	61	60	658	780	3.3	85/33
S1	61.0	3.0	13.5	14.0									8.0	0.5			1.522	60	85	585	780	2.6	73/31
S2	48.4		12.5	9.8			24.0					2.4	2.4	0.5			1.574	57	79	680	790	3.2	60/25
S3	49.0		6.0	5.2			36.0					3.4		0.5			1.604	44	85	750	820	3.5	66/27
S4	61.4	1.6	13.5	13.0									10.0	0.5			1.523	51	93	590	660	2.7	64/26
S5	41.0		6.8	3.3			44.0					4.4		0.5			1.616	37	85	760	810	3.6	56/23
S6	64.0	3.0	14.4	15.0									2.6	0.5	0.5		1.520	52	87	580	630	2.6	57/24
S7	30.0	5.0	11.5		2.2		48.0						2.3	0.5	0.5		1.613	37	83	585	640	3.5	58/24
S8	72.0		14.0	9.0			4.0							0.5	0.5		1.515	55	90	610	700	2.6	67/28
S9	71.0	1.5	5.0	16.8	5.2									0.5			1.513	60	93	605	690	2.6	68/29
SAlS1	80	8	12														1.500	52	79	550	620	2.8	55/23
SAlS2	60	20	20														1.550	50	80	600	670	2.7	56/24
SAlS3	40	20	40														1.600	55	80	600	675	2.6	57/24
SBS1 (clad)	65.0	3.0	14.0	15.0									3.0				1.519	60	87	500	560	2.7	55/23
SK12	60.0						37.0										1.583	62	64	550	610	3.0	66/27
SLS1 (core)	55.0			15.0				6.0	18.0	5.0	1.0						1.545	64	15	479	590	2.7	57/24
SLS2 (clad)	65.0			17.0				4.0	7.0	5.0	1.0						1.528	61	14	495	610	2.7	55/23
SLBS1 (core)	55.0		10.0	15.0				6.0	8.0	5.0	1.0						1.533	68	15	470	590	2.7	55/23
SLeS1 (core)	46.0			3.0	6.0	45.0											1.625	59	85	520	590	3.6	60/25

SK5-B2. Triple and quadruple optical fibres were manufactured of the following glass sets: BF8-BLF1-F2-S6, BF8-BLF5-F2-S8, BLF1-BLF2-S4, F2-BLF2-S6, F2-S6-S7-S8, SK12-BLF2-S9.

The best high silica optical fibres measured had the tensile strength exceeding 6 GPa (not presented in Fig. 3a). Figure 3a shows statistical distribution of the strength. The curves were chosen among many more measured as typical representatives of fibres from different well-known manufacturers. On the average the high silica fibres were expected to have the strength exceeding 4.5 GPa. The best multicomponent glass optical fibres had the tensile strength exceeding 3.5 GPA (not presented in Fig. 3b). On the average, however, most of the tailored optical fibres were expected to have the strength exceeding 2 GPa. This expected result is slightly more than two times less than for high silica ones. Taking bigger group of measured fibres into account this result was worse and equal to three. The measurements on big number of samples of various multicomponent glass optical fibres revealed that the statistical average strength is three times less than for high silica fibres. Narrowing the pool of data changes this result considerably as the dissipation of strength depends on the modifying ions in the multicomponent glass, what is clearly visible in Fig. 3b. The fibres designated as F21 and its stronger version F10 of the same chemical composition – SLS1–SLS2 will be used as the reference ones throughout this work for the rest of tailored optical fibres. The fibres F81–F84 were made of SLS, SBS and SLBS glasses. The fibres F85–F811 were made of B, BF, BLF and BK glasses. The fibres F812–F817 were made of K and S glasses. The fibres F818–F820 were made of high lead, SLeS and F glasses. Several dependencies between glass composition and fibre strength were observed for different glass groups.

The bigger is the contents of glass making ions (SiO_2, B_2O_3, GeO_2, P_2O_5, As_2O_3, Sb_2O_3) the stronger is the output fibre, in terms of spontaneous forming of surface micro-cracks. Lead ions in PbO oxide form (which have intermediate characteristics between network former and network modifier) generally decrease the strengths of any fibre (due to their dimensions). The effect of other intermediate oxides like Al_2O_3, ZrO_2, BeO, TiO_2, MgO, ZnO, on fibre strength is mainly neutral in the range of not too big content. The effect of network modifiers (alkali and alkali earth metal) such as CaO, BaO, Li_2O, Na_2O, K_2O, Cs_2O on fibre strength is usually negative even for intermediate content. Proportionally to the concentration of metallic modifying ions MeO + Me_2O the Si-O skeleton loosens what results in dramatic decrease in fibre strength. To maintain acceptable fibre strength in lead glass fibres (needed for very high value of numerical aperture), say with the strength above 1.5 GPa, it turned out from the measurements that the glass should contain at least 50% of SiO_2 and the alkali ions should be kept below 20%. Other glass-making ions should not cross over 10% confinement. The strengths of zirconium glass fibres (S) depended on the thermal history

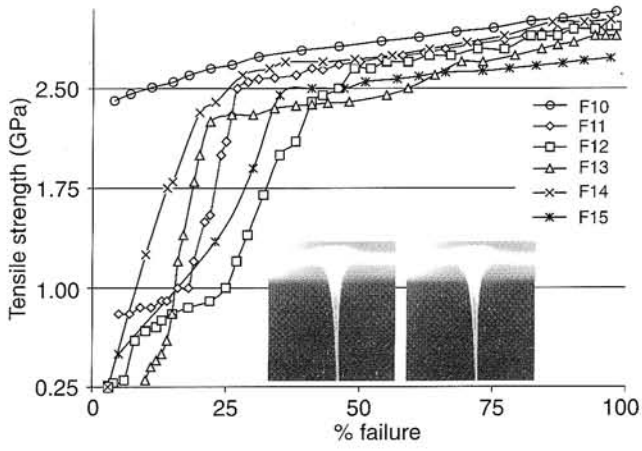

Fig. 4. Effect of outflow meniscus shape (and meniscus cooling-heating conditions) during the pulling process on MMC fibre strength. F10 – reference SBS glass optical fibre. All fibres double coated with acrylic lacquer. The meniscus changes slightly from well-defined concave (left inset) to slightly concave-convex (right inset). Insets show shapes of outflow menisci photographed from the bottom side of the crucible stack and the furnace.

after pulling what may indicate some tendency of these fibres to devitrify after a heating and cooling cycle.

Technological practice shows that the quality of MMC optical fibres depends very much on the stability of pulling conditions [4]. We measured the impact of two basic technological parameters associated directly with the pulling process: shape and cooling-heating conditions of crucible outflow meniscus and outside fibre diameter stabilisation. The insets in Fig. 4 show two basic shapes of maintained menisci: concave and concave-convex. Both types of menisci were observed at the constant speed of fibre pulling with fibre diameter not being additionally stabilised. The meniscus shape was changed by temperature of the crucible nozzle region. It was observed, during the process, that the shape did not depend on the nozzle geometry and the speed of fibre pulling but on the local viscosity and surface tension of pulled glass filament. The weak concave-convex meniscus was observed to be less stable dimensionally than the concave one. Figure 4 presents the results of measurements of tensile strengths of optical fibres pulled for continuously changing the shape of outflow meniscus from well defined concave (reference fibre F10 with stabilised outside diameter) to weakly concave-convex (fibres from F11 through F15). The result is that the geometrical spectrum of surface flaws broadens especially in the big dimension region. Then the strengths gets similar (fibre F14), with the exception of convex-concave fibre F15 which always had smaller strength. Going deeper into the convex-concave region weakens the resulting fibre. It is also more difficult for the upper boundary of this region to keep the outside diameter of fibre within ±10 μm fluctuations. After that the meniscus turns fully concave, and fibre pulling stability dramatically decreases. The tensile strength of fibres pulled for

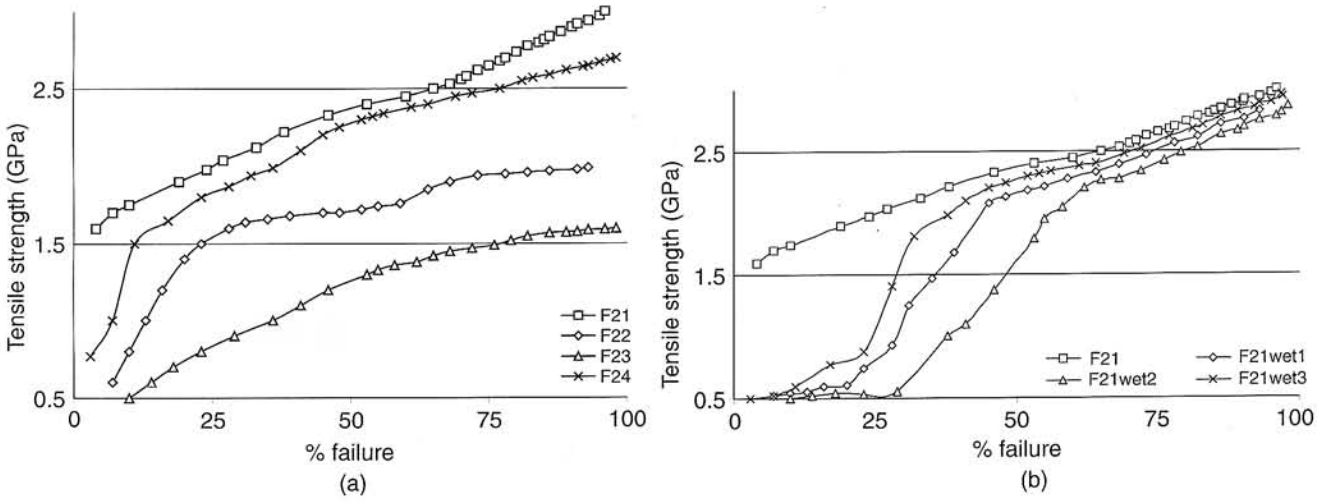

Fig. 5. Tensile strength distribution of SLS optical fibres drawn in MMC process. (a) Dependence on batch glass quality. Batch glass data gathered in Table 2. F21 – reference fibre of the highest quality, F22 – Class A glass, F23 – Class B glass, and F24 – Class C glass. (b) Batch glass of the highest quality. Dependence on batch glass wetness. The wetness was controlled from a few to a few hundred ppm in input oxides for glass synthesis.

well-defined convex-concave region did not exceed 0.75 GPa (not shown in figure). The data in Fig. 4 concern a single technological process with changing parameters, i.e., all measured fibres were the same as the reference one F10. The fibre F10 was pulled during the optimal process conditions.

Speed stabilisation leads, in the case of technological set up used in this work, to fibre diameter fluctuations closed approximately within ±10% extent. These fluctuations depended on fibre diameter and, in the case of 125 μm fibre, were ±10 μm. Our experience shows that this value is a characteristic parameter of technological set up and depends on temperature stabilisation and its distribution homogeneity in the furnace and other parameters like kind of laminar flow of argon inside the furnace, internal and external vibrations, etc. The dependence of fibre tensile strength on the stabilisation of its outside diameter during the pulling process was measured. The reference SLS glass fibre F10 was pulled using the best available stabilisation of the diameter within ±0.3 μm. The feedback stabiliser was set to ±0.6 μm, ±1.0 μm, during the same process, and eventually switched off. Statistically, the fibres with less diameter stabilisation were weaker than those with good diameter stabilisation. This, possibly, can be explained by additional outflow meniscus stabilisation through the thermal and mechanical feed backs. It also shows that the shape of the meniscus changes when a constant speed of pulling is kept.

SBS optical fibres were manufactured of batch glasses of different standard quality. Glass quality is measured by the acceptable level of impurities, tolerances of optical properties, presence of striae, optical homogeneity, bubble

Table 2. Optical fibre, compound glass quality classes.

Parameter/class/value	Ultimate (fibres F10, F21)	Class A (fibre F22)	Class B (fibre F23)	Class C (fibre F24)
Transition metal ion levels, Cr, Fe, Cu, (ppm)	0.05, 0.05, 0.05	0.05, 0.1, 0.05	0.1, 0.5, 0.1	0.2, 1.0, 0.2
Resulting fibre attenuation (dB/km) for λ = 0.8–0.9 μm	4	20	100	300
Tolerances of optical properties. Maximum variations of n_d and ν_d values	"Grade 1", $\Delta n_d < \pm 0.0002$, $\Delta \nu_d < \pm 0.2\%$,	"Grade 2", $\Delta n_d < \pm 0.0003$, $\Delta \nu_d < \pm 0.3\%$,	"Grade 3", $\Delta n_d < \pm 0.0005$, $\Delta \nu_d < \pm 0.5\%$,	"Grade 4", $\Delta n_d < \pm 0.001$, $\Delta \nu_d < \pm 1.0\%$,
Striae and inclusions	"Precision quality", P class, NVS class, not present,	"Normal quality", Striae nearly invisible,	"O class", Fine parallel striae,	"Out of striae selection", Visible striae,
Optical homogeneity. Maximum variation of n_d value	"Group H4", $\Delta n_d < \pm 1 \times 10^{-6}$.	"Group H2", $\Delta n_d < \pm 5 \times 10^{-6}$.	"Group H1", $\Delta n_d < \pm 2 \times 10^{-5}$.	"Group LH1", $\Delta n_d < \pm 1 \times 10^{-4}$.
Stress birefringence (nm/cm)	4	10	20	20
Bubble class (blisters) (mm^2) per 100 cm^3 of glass	B0, 0–0.029	B1, 0.03–0.1	B2, 0.11–0.25	B3, 0.26–0.50
Supply form	Slabs	Slabs	Slabs or pressings	Blocks or gobs

Table 3. Determined experimentally, required minimum preheating time (in min) for MMC process fibres of the highest tensile strength.

Form of supply Fibre material	Glass powder	Thick ground glass	Broken glass pieces	Fire polished glass balls	Slabs, strips, rods
SBS and GeBSi	90	80	70	65	45–60
SLS	100	80	75	65	50–60
SaIS	100	85	75	65	55–65
SleS	110	90	75	70	65–70

class, etc. The F21 fibre was used as a reference. The F21 fibre SLS glass was of the highest available quality from the best manufacturer. The parameters were in agreement with the international standards [12] and gathered in Table 2 for the measured fibre samples presented in Fig. 6:

- transition metal ion (Fe, Co,Cr, Cu, etc.) levels measured in oxide content in ppm or ppb, class of the coefficient of absorption,
- tolerances of optical properties measured in deviations of individual melt data from optical values stated in the glass description, accepted classes are called glass grades,
- striae, blisters and inclusions; localised transparent, usually thread like inclusions in glass that differ only little in composition from the base glass,
- optical homogeneity is a degree to which the refractive index varies within a glass blank or melt,
- stress birefringence, measured as a difference in optical path and stated in nm/cm,
- bubble class (category and class of blisters), bubble content of a glass is characterised by total bubble area in mm^2 per 100 cm^3 of glass,
- wet/dry class. All the above classes and especially the lower ones like C in Table 2 can be wet. Dry glasses of fibre optic grade should, at least theoretically, not contain more water at all, because hydronium ions increase attenuation at OH-Si vibration sub-bands and may decrease substantially the fibre tensile strength.

The results presented in Fig. 5a show statistically how the bulk glass quality may influence the tensile strength of the resulting optical fibre. The measurements were also performed for other glasses like SBS, SAlS, also some flints and crowns of optical purity. The results were always the same qualitatively (worse glasses gave weaker fibres) but quantitatively the results depended very strongly on the glass family and dominating ion system. For example, fibres made of some glasses exhibited anomalously decreased or increased tensile strength as a function of impurity content. The combination of some ions in compound glass may decrease the fibre strength considerably.

The content of water, originating from the input oxides, also was observed to decrease the fibre strength. The results of such measurements were presented in Fig. 5b. The fibre F21 was used as a reference. The fibres F21wet were pulled from the same glass system of the same class quality, but the SLS glass for the fibre was prepared using more and more wet input oxides (approximately contained 5 ppm for dry case and respectively 100 ppm, 300 ppm and 700 ppm of OH^- for wet cases). The tensile strength of the resulting fibre does not change considerably for bigger loads. The result of water is seen clearly for smaller loads, i.e., for bigger faults. The high temperature pulling process is not able to remove fully the effect of wet oxides. Possibly the process lasts too short, for the chemically bound water to be removed totally.

To check the influence of glass degasification and homogenisation in the crucible we applied different time periods of crucible heating, slightly above the threshold of glass melting, before the beginning of fibre pulling. The time of heating was 15, 30, 45, 90 and 105 minutes. This time was measured from the point when the temperature of furnace-crucible system was high enough and the glass viscosity reached appropriate level for fibre pulling. Thus, this was additional time of melt preheating. To prevent the glass from flowing down, the nozzle was closed. SBS optical fibres were manufactured, doubly covered with protective lacquer. The glass was fed to the crucibles on the batch basis (not continuously) in small slabs (platelets). The F21 fibre was used again as a reference. The changes between 105- and 90-minute cases were virtually not measurable, for this particular process and SBS glass. The remaining cases were: 45 min, 30 min, and 15 min. The measurements indicate that the saturation time was here around 55–60 minutes. This defines, for this case, the minimum time required for crucible system preheating. The process preheating time was observed, on the basis of numerous measurements for other process parameters, to be a function of input glass forms and kind of glass supply. The minimum preheating time is defined here as necessary waiting period in the high temperature to obtain a fibre of the best available, in such circumstances, tensile strength. The required preheating time is quite long, what was gathered in Table 3. Originally we expected this time to be shorter. Several basic forms of batch supply were applied: glass powder, fire polished glass balls, slabs, platelets, strips, rods, broken glass. Unworked surfaces or broken ends introduced more gas bubbles into the melt what required more preheating time to obtain optical fibre of tensile strength comparable to that of the reference fibre F21. It was also noticed that, in case of wet batch glass, the longer preheating time led to stronger optical fibres.

The crucible-furnace system behaves quite differently in the case of continuous glass feeding. The furnace was

equipped with mechanised throughputs for several glass rods to be fed continuously to the core and cladding crucibles [3]. The rate of feeding could be adjusted so as not to change the relative levels of glass melt in crucibles during the pulling process. The rods of different diameters were fed in the crucibles to force the feeding system to work at considerably different speed to maintain the constant glass melt level. At some rod-diameter and feeding-speed combination we observed bubble trapping effect into the melt what immediately resulted in weaker optical fibres manufactured during this part of the process. It gives a measurement-supported evidence that, even in the case of continuous glass feeding to the crucibles, some preheating time is required, to obtain the fibres of the highest tensile strength. Here, however, the preheating time has a different technical meaning, because in the case of continuous feeding we cannot close the nozzle as in the batch load case. Preheating time is set here dynamically through the optimised choice of the rate of feeding. The confinements are imposed on the minimum crucible capacity, minimum height of glass melt and maximum rate of rod feeding. These parameters are chosen experimentally for the particular process.

The quality of the atmosphere in the furnace was observed to have a considerable impact on the fibre strength. Factors influencing the atmosphere quality are: cleanliness, humidity, homogeneity of thermal field distribution, laminar flow of insulating gas, etc. The furnace is sealed and clean argon circulation system, with laminar flow around the crucible stack and nozzles, is applied. The heaters are sealed in the furnace, the internal ceramic screens polished to minimise dust generation. Dust particles in the furnace are kept to very low levels due to the circulation effect of clean insulating gas. Slight overpressure is maintained in the furnace chamber. Depressurising the chamber, unsealing, or moving the process to the furnace with unpolished ceramic screens allows us to control the level of internal dust pollution. Fine particles are generated always inside the hot furnace from the refractory materials. The density of particles increases with the temperature. Our own measurements indicate that the typical density is in the range of 1–10 particles per litre, and depend on the subtleties of furnace construction and maintenance of clean gas system. The sizes of these particles vary statistically in the range 0.3–1 μm. These particles cause a considerable reduction of the tensile strength of the fibre. It was found experimentally that the tensile strength of optical fibre is dependent on the particle presence and parameters through the following approximate and simple relation: $s_t = (r_p)^{-1/2}$, where s_t is the tensile strength of fibre, r_p is the dust particle radius. The effect of dust influence on fibre strength and dust reduction through flowing of filtered gas into the furnace and closing the furnace by using a clean gas shutter was investigated. Introducing into the furnace, with the gas, a controlled pollutant of very narrow spectrum of sub-micrometer particles (0.2–0.3 μm) resulted in comparatively narrow strength distribution. The distribution of dust particle diameters for the next case was 0.3–1.0 μm what immediately broadened the strength characteristic. Increasing the density of dust particles and their dimensional spectrum quickly leads to unacceptable levels of tensile strength of manufactured fibres. Just switching off the argon gas system and opening the furnace to the laboratory environment (which was of the cleanliness class of the order of 10000) led, in our case, to fibres which never had better tensile strength than 1 GPa, regardless of glass grade, even after careful double coating.

Humidity affects the fibre strength in many different ways. At some stage of the flaw building process the presence of water is necessary at the flaw tip [6,7]. Water (or OH- ions) is present nearly everywhere: in glass substrates (oxides) and in the hot furnace atmosphere. It is necessary to flow dry gas through, to prevent the presence of water in the furnace. The effects of the humid atmosphere in the MMC furnace on fibre strength were investigated. Three fibres were drawn when argon saturated, not saturated with water vapour and dry was flown through the furnace. The fibres were pulled from the Class B glass. It is necessary to maintain a flow of dried gas in the furnace for making high-strength fibres. The first order effect of water is that the hydroxyl ions introduce a lot of large dimension, surface cracks to the pulled fibre. A lot of other effects of water on fibre strength were observed like faster ageing and faster devitrification for some glasses. Some compound glasses turned out to be slightly hygroscopic [9].

The effect of water on bare and variously coated MMC fibres were measured. Humidity conditions of fibre storage were changed. Tensile strength was lowered even of two orders of magnitude for some glasses. The destruction process begins when an uncoated fibre touches the capstan reel. The flaws form immediately on the fibre surface, even if the fibre is then kept in a very dry environment. Weibull characteristics were measured for fibres covered with: a single acrylic lacquer (faster pulling process), single acrylic lacquer (slower pulling process), double acrylic lacquer, double acrylic lacquer and pressurised die for primary coating, lacquer and thick silicone jacket to 1 mm outside dimensions. The quality of the coating process and thickness of the coat (and jacket) has an essential meaning for the tensile strength of compound glass fibre. Faster coating process results, in some cases, in thinner coat layer, giving lesser strength. Different coating materials were tried. Two or three layers were applied and an elastic outside silicone jacket resulting in fibre of the outside diameter equal to 1mm. The primary coating materials were apart from acrylic lacquer: thermally cured epoxy, Urethal 450, EPI polyimide lacquer, UV cured resin, Kynar and Hytrel. Though the dependence of fibre strength on coating material was observable but it changed considerably with the fibre material. Thus, it seems that individual coating material must be chosen for particular compound glass for optimum strength performance of optical fibre. Much stronger dependence of fibre strength was observed against the coating quality, especially when the fibre was exposed for a long term to a hostile environment.

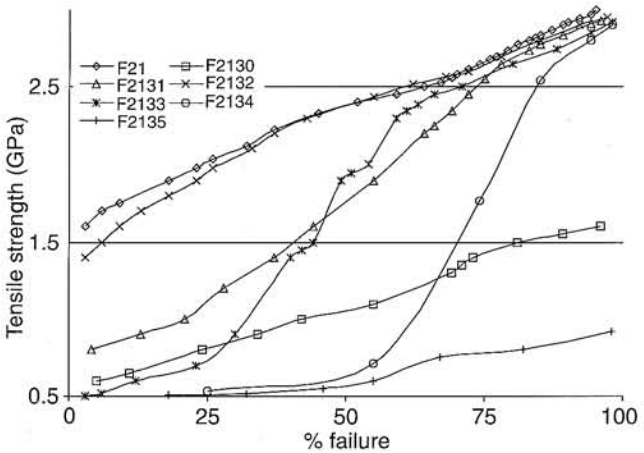

Fig. 6. The effect of moist environment and environmental changes on the tensile strength of coated and jacketed compound glass optical fibres. Degradation of the strength is a measure of the particular fibre coating system permeability to water.

The resistance of coated optical fibres were measured against water (OH- ions) permeability. Coated, compound glass, optical fibres were kept in humid environment and an impact on the tensile strength was measured. The results are presented in Fig. 6. All the samples of F21 fibre family were coated with Urethal 450 (thermally cured) and jacketed in silicone. Thermal cycles of heating (80°C) and cooling (0°C) was applied in some cases to look for pinhole effect in primary coating. There were applied 12 cycles per 24 hours. The fibre ageing hostile environment was as follows: single primary coating, week in water at room temperature (F2130); double primary coating, week in water (F2131); triple primary coating, week in water (F2132); double coated, week of thermal cycles in air and week in water (F2135); triple coated, week of thermal cycles in air and week in water (F2134); triple coated, week of thermal cycles in humid atmosphere, relative humidity h_r = 90% (F2133). The deterioration in the tensile strength of investigated optical fibres is a measure of water permeability during such tests. These investigations show directly that there are virtually no ideally sealing materials for fibre jackets and coats. Several additional methods of fibre protection against water, like cabling with water repelling agents, have to be undertaken for particular application. It was revealed that some hard coating, especially thermally cured ones, may be irresistive to thermal cycles. On the other hand, the silicone resin is quite permeable to water and the jacketing layers of considerable thickness have to be applied, to keep the fibre strength for longer time in the moist environment.

One of the basic problems of manufacturing of tailored optical fibres is the need to combine glasses of distant thermal and mechanical characteristics. The core and cladding in a fibre made of glasses of distant characteristics exhibit very interesting properties for sensory applications of such fibres [4–5]. We checked the impact of the discrepancy in some of these characteristics on the fibre tensile strength. Two families of Weibull characteristics were measured with the parameter chosen as a difference in linear expansion coefficients $\Delta\alpha$ (%) = $\alpha_{co} - \alpha_{cl}(10^{-6}°C^{-1})$ between core and cladding for two different cases when there were close softening temperatures $\Delta T_s = \left|T_s^{co} - T_s^{cl}\right| < 25°C$ and distant softening temperatures $\Delta T_s = \left|T_s^{co} - T_s^{cl}\right| > 50°C$ between core and cladding glasses. The results of measurements were presented in Fig. 7. For very close softening points of core and cladding glasses the difference in linear expansion coefficients were 25, 20, 15, 10, 5, –5, –10, –15, –20, and –25%. These values correspond accordingly to the

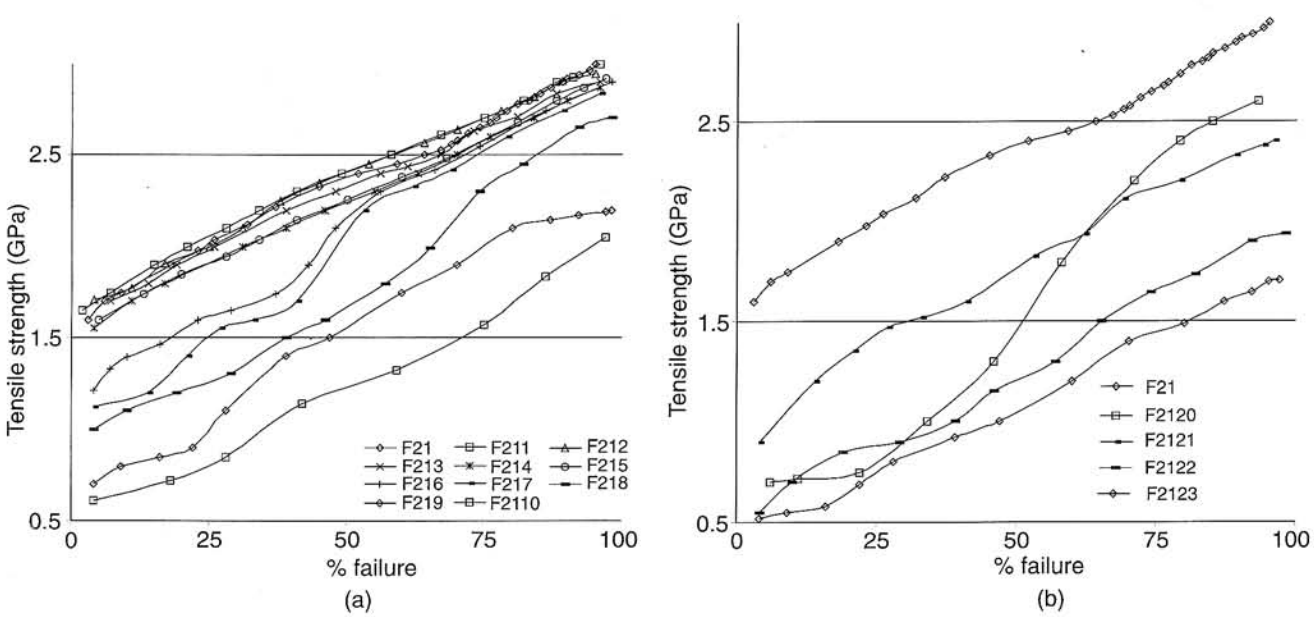

Fig. 7. The effect of differences in linear expansion coefficients in core-cladding glass system on the tensile strength of resulting optical fibre: (a) close softening points of core and cladding glasses and (b) distant softening points of core and cladding glasses.

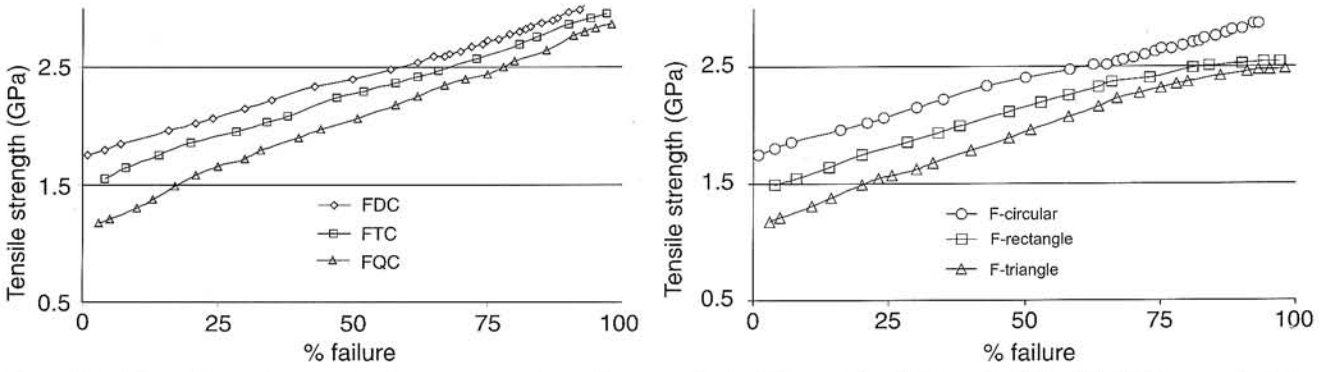

Fig. 8. The effect of internal, geometrical, structure on the tensile strength of multi-layered, tailored, optical fibre (a). FDC – core-cladding fibre, FTC – core-double-cladding fibre, FQC - core-triple-cladding fibre. The effect of internal, geometrical, structure on the tensile strength of optical fibres with different shapes of the cores (b). F-circular – circular core fibre, F-rectangle – rectangular core fibre, F-triangle – triangular core fibre. All fibres made of GeBS glass.

following fibres data presented in Fig. 7a: F2110, F219, F218, F 211, F 212, F213, F 214, F215, F216, F217. The results of measurements show that the distribution of characteristics against zero difference in linear expansion coefficients is not symmetrical. The fibres get weaker for large positive value of $\Delta\alpha$ and stronger for moderate negative value of $\Delta\alpha$. It seems that moderate compressing action exerted by the core on the cladding has the effect on fibre strengthening. At distant values of the softening points between core and cladding glasses the difference of linear expansion coefficients has greater meaning and the fibre gets much weaker. The results presented in Fig. 7b are: F2120 – $\Delta\alpha$ = –20%, F2121 – $\Delta\alpha$ = –15%, F2122 – $\Delta\alpha$ = +15%, F2123 – $\Delta\alpha$ = +20%. Internal stress within the fibre tends to put the core region in tension and the outer surface in compression, thus improving the strength. This process is, however, highly nonsymmetrical. One can say that the aggregated technological length of the set of two glasses, creating a fibre, is not only the function of differences of particular temperatures within these glasses but also strongly depends on the value and sign of relevant parameter discrepancies. An aggregated technological length is here an important fibre making parameter, because it defines, through a single value, the material set compliance for the manufacturing of, mechanically stable, glass filament. The results of these investigations, and the like analysing sub-

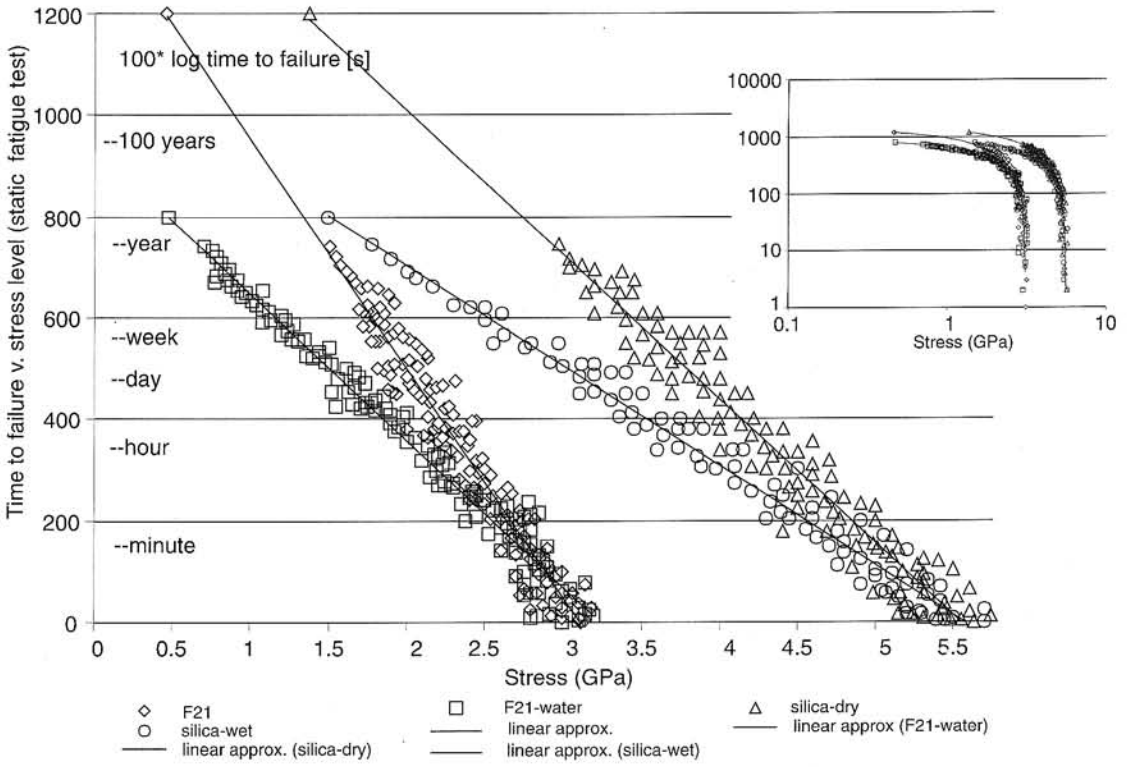

Fig. 9. Time to failure versus stress level characteristics of high silica and SLS optical fibres. Fibres measured in dry air and in water. The inset shows the characteristics in the logarithmic coordinates.

tleties of allowable characteristics discrepancies, shed a new light on the manufacturing possibilities of optical fibres from quite distant materials.

Tensile strength of an optical fibre is expected to depend on the internal structure of the fibre. To check that dependence experimentally we measured a few multi-layer fibres made of the same glass system (BF8-BLF1-F2-S6) but with different geometrical structure. Three fibres were measured: double crucible (FDC), triple crucible (FTC) and quadruple crucible (QTC). Proportions of the successive layers in these fibres were 1:1:1:1. The measurements results in the form of Weibull characteristics were presented in Fig. 8a. The differences in the tensile strengths are not very big. Though it seems that more complex internal structure of the fibre leads to some diminishing of the strength. Further investigations and measurements show that the tensile strength of tailored optical fibres of complex internal structure depends very much on the discrepancies of thermal and mechanical properties of glasses used for successive fibre layers. This dependency is much stronger than for a classical single clad compound glass optical fibre. Thus, the individual glass characteristics in multi-clad optical fibre have to be chosen more precisely and within smaller range of parameters. The increase in fibre internal complexity narrows the choice of materials and vice versa.

Weibull characteristics of strength distribution were investigated for tailored optical fibres of different geometrical shapes of the core. Three fibres were measured of the same glass composition GeBS but of circular, square and triangular cores. The results of measurements are presented in Fig. 8b. All the fibres had nearly the same internal geometrical proportions 125/65 μm. The triangular and square core fibres, had quite sharp corners of the cores. The diameter of corner bending was around 10 μm. Other families of triangular and square fibres were pulled with different value of the corner diameter up to 20 μm. The corner diameter was set by the nozzle construction of the crucibles and process parameters [4]. The fibres with blunt corners had bigger strength approaching that of circular core one. Sharpening of the corners is done at the cost of complications introduced into the pulling process. Much more complex pile of crucibles with shaped nozzles and inter-nozzle diaphragms must be introduced. The process must go on in different temperature. These changes seem to influence the resulting fibre strength, as it is presented in Fig. 8b.

Time to failure versus stress level characteristics are the results of static fatigue tests. The static fatigue tests are arduous and time consuming, thus a special multi-fibre stress test stand was build in which we were able to measure several tens of samples at a time. These static characteristics supplement the dynamically measured Weibull tensile strength distributions. Figure 9 presents the results of measurements of two kinds of optical fibres: high silica telecommunication multimode and reference fibre F21 of SLS compound glass. Both fibres had the best primary coats and silicone jacket up to 1 mm in outside diameter. Two sets of

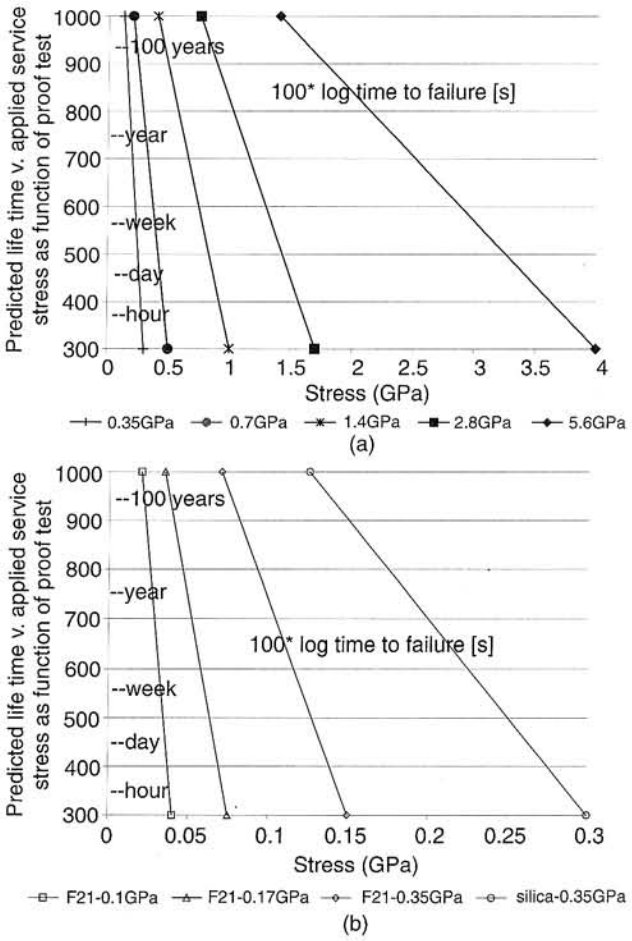

Fig. 10. Predicted lifetime versus applied service stress as a function of the level of proof test: (a) high silica fibres used as a reference, (b) compound glass fibres used as a reference.

pairs of fibres were measured, one pair was kept in dry environment with relative humidity of 55% at room temperature, while the second pair was submerged in water. The inset shows the same characteristics but in logarithmic coordinates. Water changes the slope of the linear approximations to the collection of measured data points. The slopes are a direct measure of fibre resistance to ageing, through mechanical fatigue. The mechanical fatigue is enhanced considerably by the long-term influence of water. For short times the influence of water, for properly coated and jacketed fibres seems not to have a meaningful effect but as time goes on, water impact gets dominant. The coats and jackets simply leak.

Predicted, optical fibre, lifetime versus applied service stress characteristics shows Fig. 10. An optical fibre is expected to work under the same service stress. This stress should be zero but practically, due either to fibre cabling or to the real work conditions, it is nonzero. The measurements of fibres in different working conditions allow to estimate quite precisely the service stress. We investigated the influence of experimentally assumed service stress on the predicted lifetime of the fibre, which undergone certain level of proof test. Figure 10a shows the characteristics of the same high silica reference fibre which was investigated in Fig. 9.

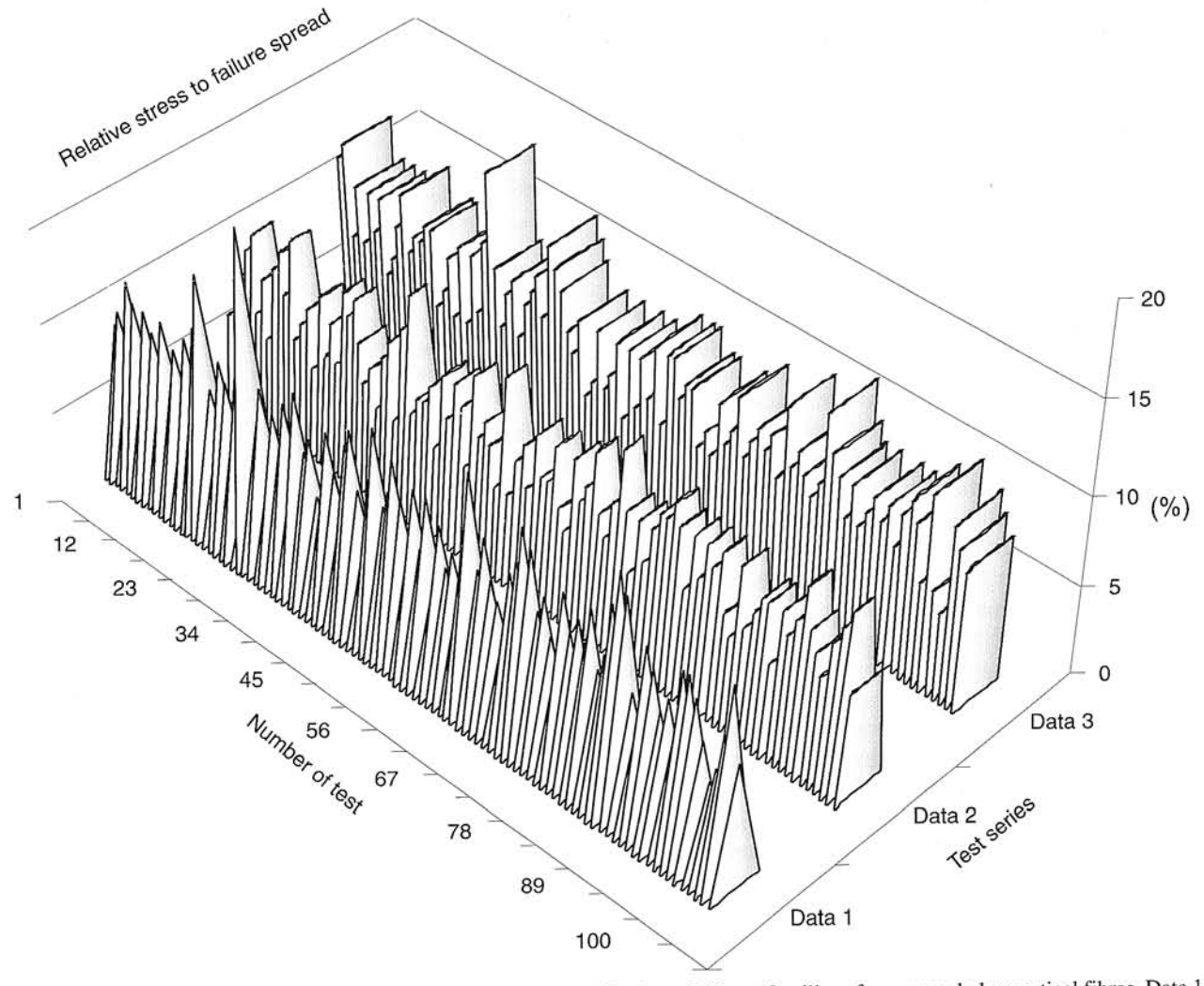

Fig. 11. Relative tensile strength (stress to failure) spreads measured for three different families of compound glass optical fibres. Data 1 – SLS glass optical fibres, Data 2 – SBS glass fibres, Data 3 – GeBSi glass fibres.

The best high silica optical fibres of short length exhibited tensile strength over 6 GPa. These were proof tested with the load of 5.6 GPa. The expected lifetime for such fibres (kept in dry environment) is 25 years under the service load of over 2 GPa. These results show the potential margin of application safety. Figure 10b shows expected life time characteristics for the reference fibre F21 after proof testing the whole measured length of this fibre with the following proof loads 0.35 GPa, 0.17 GPa, and 0.1 GPa. The measurement results show that the fibre F21 can work for 25 years under the constant service load of 0.1 GPa when tested with the proof load of 0.35 GPa. Comparing these results with the silica fibre one obtains from the measurements in the same conditions the value of the service load equal slightly below 0.2 GPa or only two times more than for F21. It shows that the quality of fibre F21 is quite good, taking into account the very distant material characteristics of both fibres. The domination of high silica fibre reveals itself at much higher levels of proof-test loads. Some of these loads are unavailable for F21 fibre. However, we have also proof tested the F21 fibre with the load of 1.4 GPa (not shown in the figure). The measurements have shown that the long lengths of more than 500 m have withstood this load successfully, rising the expectations for service load to over 0.5 GPa for the standard lifetime of over 25 years. These results show indirectly the potential of application of tailored optical fibres with high NA, and small outside dimensions and engineered environmental sensitivities, in the smart structures and skins. The results of measurements allow one to draw a rule of thumb for the needed levels of prooftesting. Since some technical data were assumed as the fibre and fibre cable quality standards like: expected lifetime of 25 years and 0.1 GPa as the service stress, also 0.35 GPa was assumed as a required value of proof testing level [12]. A rule of thumb says that the proof test should be at least three times bigger than the maximum expected optical fibre services stress.

To support directly the validity of statistical measurements done in this work we performed a number of measurements in each described category and investigated the relative stress to failure spread. $(STFS)_r$ was defined as a difference between maximal and minimal tensile strengths for a particular specimen of fibre related to the median tensile strength of the same fibre. Only the best fibres were taken into account, with the characteristics without weak tails caused by either the deficiencies of material or technology. For a strong fibre, the prevalent flaw sizes (governing the fibre strength) have a very small spread from a mean size, which can be seen from dynamic load test results. The stress to failure spread is considerably under 10% in the best high silica fibres. The relative stress to failure spread measurements for three different families of high quality tailored optical fibres were presented in Fig. 11. Since these measurements are basing on hundreds of individual measurements and some of them are comparable to the results for the best fibres presented in Figs. 2–10 the conclusions may be drawn about the stability of the MMC technology of tailored, compound glass optical fibres. The histograms of the measurement results presented in Fig. 10 were extracted. These histograms of tensile strength spread display the distribution of cases in terms of the value of relative stress spread against the frequency of these cases. Over 90% of cases are confined between the stress spread values of 5–11%. And the extreme value was never bigger than 14%. These results show the maturity of the technology.

4. Conclusions

Our investigations of bare fibres indicate that a large number of surface cracks builds up, even without any stress exerted on the fibre just a few minutes or hours after the pulling, what depends on environmental moisture. But after this initial process the cracks seem not to build up. Crack propagation resumes when the stress is applied. Thus, we noticed, that at very low stresses or no stresses at all, water plays an important role in the initial crack building. Stressing the bare fibre in water environment shows much faster crack propagation (much shorter life times of fibres) than in dry environment. Adding to the water some base (like 5% NaOH) reduced the average lifetime of typical compound glass fibre further of more than 50% in comparison with the survival time in distilled water environment. An acidic and basic environment is seen to influence the crack growth. Silica itself and most silica glasses are weakly acidic compounds. The fibre lifetime in water is shorter than in air, thus, it is possible that the glass reacts with water to form a silicic acid.

Comparison between the calculated values of critical crack sizes and experiment data for average tensile strength of high-silica and compound glass fibres reveals that the crack dimensions in the latter are nearly an order of magnitude bigger (single nm against tens of nm).

Comparison between the cyclic and permanent fatigue loads on the bare fibre in dry air shows that the lifetime depends on the magnitude and total duration of the applied stress, but not on its cyclic rate. This shows that the cracks build up always when the stress is exerted and do not heal during the stress breaks. Fatigue and ageing effects of the fibre are associated with the propagation of sub-critical surface cracks under load. Comparison between the cyclic and permanent fatigue loads on the coated and jacketed fibre in wet environment indicates the formation of pinholes in the coating through which the environmental moisture penetrates the fibre. The fatigue results for cyclic loads are statistically worse than static ones.

The results that were obtained for the fibre groups of different linear expansion coefficients between the core and cladding show the impact of internal stresses in the fibre on the strength. With this value negative the strength of the fibre is increased. The internal stress within the fibre, which tends to put the core region in tension and the outer surface in compression, improves the strength.

The minimum lifetime of a fibre under load can be estimated from the dynamic fatigue measurements and by determining the low strength confinement through the proof test. The dynamic fatigue measurements give median strength and standard deviation for a constant stress rate. Increasing the stress rate considerably and thus diminishing the time of measurement (and performing the measurements in very dry environment) one is arriving at an inert strength of the optical fibre. The inert strength is a direct measure of the initial flaw size (which had no time to propagate). These data allow one to determine the Weibull parameters and a minimum lifetime after certain level of proof test. It is also possible to determine the required proof stress to guarantee a minimum lifetime. When one assumes a given level of proof-test stress, it is possible to calculate the allowable long-term service stress for a given minimum lifetime in service. For example, assuming the lifetime of 25 years, under a constant service stress of 1 GPa, the measured optical fibre would have to be proof tested at a level of about 2.5 GPa or approximately 2.5 times the assumed service stress. This factor changes approximately from 2 to 3.5 and can be safely assumed for most applications as 3.0.

The measurements of compound glass optical fibres of more complex internal structure show that there is some dependence of the tensile strength on this structure. Generally, the internal structures, of the mechanical-thermal behaviour compressing the outer regions of the fibre, may increase the strength. The internal structures of very complex shapes, especially the ones involving corners and sharp details, may weaken the strength. This impact was not observed to be bigger, for customary tailored optical fibres than, ±10–20%. Exotic core shapes like strip (actually, 1:10 ellipsis) or multi-core (with cores laid close to the fibre outer boundary) caused the fibre strength to be lowered by 40–70%, when compared with a classical fibre of the same material.

The design of particular fibre characteristics, especially the guided-wave sensitivities to the external world is of ut-

most importance for the research of new constructions of optical fibre sensors. Special characteristics are very frequently obtained in tailored optical fibres made of distant glasses. The glasses are distant in terms of thermal, mechanical, optical and, generally, physic-chemical characteristics. It was showed, that in the case of close softening points of constituent glasses, we are able to combine even distant materials in a single optical fibre, and the strength of such a structure can be acceptable.

The results of systematic measurements published in this work are showing directly the influence of the MMC process parameters and material quality on the tensile strength of optical fibres. With all the material and technological deficiencies removed one can arrive at the reasonable levels (from the point of view of practical applications) of the strength for compound glass optical fibres. So far the tensile strength of these fibres was one of the factors confining the wider applications. Perhaps these investigations, will lead to the increased interest in tailored optical fibres, as potential components for photonic functional devices and sensors.

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References

1. J. Dorosz and R. Romaniuk, "Optical fibre technology by triple and quadruple crucible methods", *Proc. 6th International Fibre Optics and Communications Conference FOC'82*, Los Angeles, 99–102 (1982).
2. W. Leś, J. Dorosz, and R. Romaniuk, "Research-manufacturing department of fibre optics", *Szkło i Ceramika* **34**, 145–148 (1983).
3. J. Dorosz and R. Romaniuk, "Fibre Optic Department of Biaglass Co. Twenty years of research activities", *Optica Appl.* **37**, 267–291 (1998).
4. J. Dorosz and R. Romaniuk, Multicrucible technology of tailored optical fibres, *Optica App.* **37**, 293–322 (1998).
5. J. Dorosz and R. Romaniuk, "Soft glass optical fibres", Research-Manufacturing Department of Fibre Optics, Glass Works "Biaglass", *Application Notes and Product Catalogue*, Warsaw-Białystok, 1999.
6. *Optical Fibre Telecommunications II*, edited by S.E. Miller and I.P. Kaminow, Academic Press, Boston, 1992.
7. J.C. Daly, *Fibre Optics*, CRC Press, Boca Raton, 1994.
8. T. Izawa and S. Sudo, *Optical Fibres: Materials and Fabrication*, KTK Sci. Publ., Tokyo, 1987.
9. *Optical Glass and Fibre Optics Catalogues*, 1980–1999.
10. A.A. Griffith, "The phenomena of rupture and flow in solids", *Philosophical Transactions of the Royal Society* **A221**, 163 (1920).
11. W. Weibull, "A statistical distribution function of wide applicability", *J. Appl. Mech.* **18**, 293 (1951).
12. *ISO, DIN and ANSI Standards for optical grade glasses and telecommunication optical fibres*, 1980–1998.
13. "Optical fibres and their applications", edited by J. Dorosz and R. Romaniuk, *Proc. SPIE* **3731** (1998).

