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Erbium-doped aluminophosphosilicate optical fibres

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***Abstract*.** **We have studied the active properties of erbium-doped aluminophosphosilicate (APS) core fibres in wide ranges of erbia, alumina and phosphorus pentoxide concentrations. The absorption and luminescence spectra of the P_2O_5- or Al_2O_3-enriched erbium-doped APS fibres are shown to be similar to those of the erbium-doped fibres singly doped with phosphorus pentoxide or alumina, respectively. The formation of $AlPO_4$ in APS fibres leads not only to a reduction in the refractive index of the glass but also to a marked increase in Er_2O_3 solubility in silica.**

Keywords: *active aluminophosphosilicate fibres, clusters, gain coefficient.*

1. Introduction

Rare-earth-doped optical fibres are a key component of fibre lasers and amplifiers. The solubility of rare-earth oxides in silica glass is known to be rather low, so increasing their concentration leads to clustering and substantially reduces the pump-to-signal conversion efficiency because of a number of cooperative effects [1 – 3]. To prevent clustering, the fibre core is, as a rule, additionally doped with aluminium oxide or phosphorus pentoxide. For this approach to be effective, silica glass should contain at least 10 Al or 15 P atoms per rare-earth ion [4]. Since doping with phosphorus pentoxide (alumina) increases the refractive index of silica glass, raising the rare-earth concentration in the fibre core increases the core – cladding index difference, Δn.

Recent years have seen increasing interest in high average or peak power fibre lasers and amplifiers [5 – 7]. Their maximum output power is limited by nonlinear effects, which can be suppressed by increasing the mode field diameter of the fibre and reducing its length. The active fibre length can be reduced by raising rare-earth concentration, but the associated increase in Δn proves unacceptable at a large mode field diameter because single-mode operation becomes disturbed. For this reason, large mode area fibres that are used in high-power lasers and amplifiers contain relatively low concentrations of rare-earth dopants.

One possible solution to this problem is to use $P_2O_5 - Al_2O_3 - SiO_2$ ternary glasses as hosts for rare-earth oxides. Aluminophosphosilicate (APS) glasses are unique in that they contain the compound $AlPO_4$, whose refractive index is lower than that of silica glass [8]. This offers the possibility of doping the fibre core with high concentrations of alumina and phosphorus pentoxide while maintaining Δn low.

On the other hand, the properties of $SiO_2 - AlPO_4$ glasses are in many respects similar to those of undoped silica glass because the Al^{3+} and P^{5+} ions in $AlPO_4$ are in fourfold coordination and substitute for two silicon atoms to form $\equiv P - O - Al \equiv$ bonds, analogues of $\equiv Si - O - Si \equiv$ [8, 9]. This raises the question of whether the aluminium and phosphorus atoms of the $AlPO_4$ groups are capable of increasing the rare-earth solubility in silica glass. Previous studies of APS fibres [10 – 15] have provided no unambiguous answer to this question.

To assess the effect of $AlPO_4$ on rare-earth solubility, we studied the active properties of erbium-doped APS core fibres in wide ranges of erbia, alumina and phosphorus pentoxide concentrations. Erbia was used as a dopant because clustering has a stronger effect on the active properties of erbium-doped fibres in comparison with the other rare earths, which facilitates determination of the erbia solubility in different hosts glasses.

2. Experimental

Several series of optical fibre preforms, differing in phosphorus pentoxide, alumina and erbia concentrations, were produced by modified chemical vapour deposition (MCVD). The process was modified so that all the dopants (including alumina and erbia) were deposited from the vapour phase. In particular, the $AlCl_3$ powder used as the aluminium precursor was placed in a container thermostated at 130 °C. The $AlCl_3$ vapour was delivered by argon carrier gas to the reaction zone through a separate heated line and was mixed with the other components inside the substrate tube. A similar procedure was employed to dope with erbia, using erbium dipivaloylmethanate, $Er(dpm)_3$, as the precursor.

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Vapour phase doping ensured accurate control of the core glass composition and high doping levels (up to 20 mol % Al_2O_3 and P_2O_5, and up to 1 mol % Er_2O_3). The phosphorus pentoxide and alumina in the core glass were determined by X-ray microanalysis on a JEOL JSM-5910LV electron microscope. The erbium concentration was determined on a CAMEBAX electron probe X-ray microanalyser. Refractive index profiles were obtained with a York Technology P102 preform analyser.

3. Experimental results and discussion

3.1 Absorption and luminescence spectra

The optical loss spectra of all the erbium-doped fibres were obtained by the standard cut-back technique. In a number of previous studies, phosphorus-enriched APS fibres were found to have a very broad (600 – 1600 nm), strong (100 – 600 dB km^{-1}) absorption band centred at 1200 nm [10 – 14]. As shown recently [16], the absorption was due to contamination of the phosphosilicate glass with divalent iron. In this study, no 1200-nm absorption was observed owing to the high purity of the precursors used and careful control of the preform fabrication process. Accordingly, all the erbium-doped APS core fibres studied had relatively low optical losses (within 40 dB km^{-1}) in the near IR away from the absorption peaks of erbium. The absorption spectra of Er^{3+} for the Al_2O_3-enriched APS cores were similar to those for aluminosilicate fibres, and the spectra for the P_2O_5-enriched APS fibres were similar to those for phosphosilicate fibres (Fig. 1a).

For all of the fibres studied, we analysed the absorption at 980 and 1530 nm in relation to the mean erbia concentration in the fibre core:

$$C_m = \frac{\int_0^\infty C(r)I(r)r\mathrm{d}r}{\int_0^\infty I(r)r\mathrm{d}r}, \quad (1)$$

where $C(r)$ and $I(r)$ are the radial distributions of the erbium concentration and electric field intensity at the wavelength of interest (980 or 1530 nm), respectively. The extinction coefficient for the absorption band centred at 1530 nm was the same in all the fibres (780 dB m^{-1} per mole percent of Er_2O_3) to within the measurement accuracy ($\sim$ 10 %). The extinction coefficient for the absorption band centred at 980 nm was 240 dB m^{-1} per mole percent of Er_2O_3 in the phosphosilicate and phosphorus-enriched APS fibres and a factor of 1.5 higher (350 dB m^{-1} per mole percent of Er_2O_3) in the aluminosilicate and aluminium-enriched APS fibres.

To measure the luminescence spectra of erbium ions in various hosts, the radiation from a 980-nm single-mode laser diode was launched into an active fibre. The erbium-doped fibre was sufficiently short to avoid reabsorption-induced distortion of the luminescence spectrum, and lasing was prevented by obliquely cleaving the output fibre end. The luminescence spectrum was measured through the lateral fibre surface using an Ando AQ6317B optical spectrum analyser. The results demonstrate that, as in the case of absorption spectra, the erbia luminescence spectra for the Al_2O_3- or P_2O_5-enriched APS fibres were similar in shape to those for the fibres singly doped with Al_2O_3 or P_2O_5, respectively (Fig. 1b).

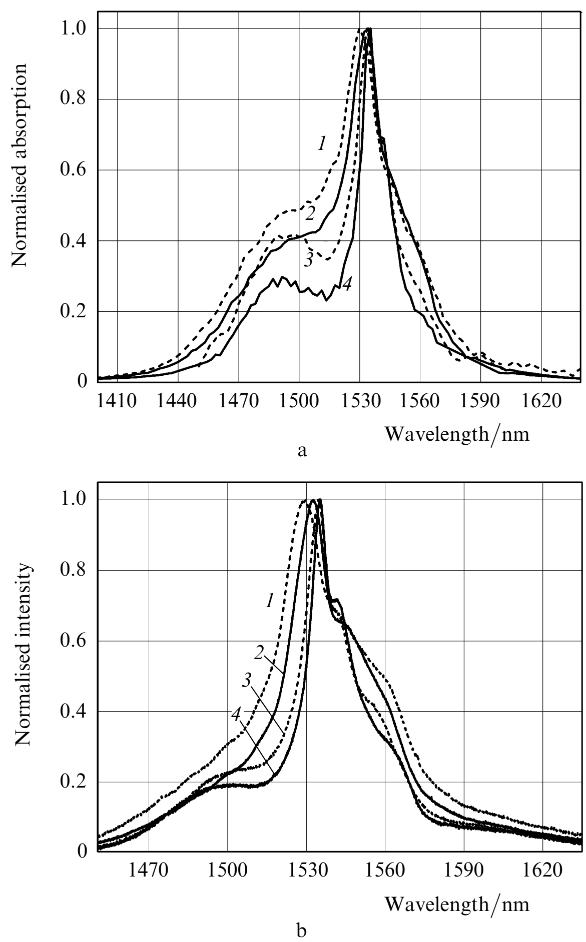

Figure 1. (a) Typical small-signal absorption spectra around 1.55 μm and (b) erbium luminescence spectra for fibre cores doped with (*1*) Al_2O_3, (*2*) Al_2O_3 + $AlPO_4$, (*3*) P_2O_5 and (*4*) P_2O_5 + $AlPO_4$.

Note that earlier studies of the UV/visible absorption spectra of passive fibres [16] and studies of ytterbium-doped fibres [13, 14] also indicated that the APS fibres were similar to aluminosilicate or phosphosilicate core fibres. The reason for this is that α-quartz and berlinite (crystalline $AlPO_4$) are similar in properties, as are therefore SiO_2-$AlPO_4$ glasses and silica glass [8, 9]. Almost all the aluminium and phosphorus atoms (except for those in excess of the equimolar ratio) are present in the form of $AlPO_4$. APS glasses can therefore be thought of as SiO_2-$AlPO_4$ glasses doped with P_2O_5 (excess phosphorus pentoxide) or Al_2O_3 (excess alumina). Thus, the luminescence spectra of APS fibres are governed by the dopant (Al_2O_3 or P_2O_5) that is present in excess.

3.2 Gain performance of erbium-doped fibres

Erbium clustering is the main factor that degrades the gain performance of erbium-doped fibres [1 – 3]. To compare erbium solubility in different glasses, the pump-to-signal power conversion efficiency was measured in a highly saturated fibre amplifier configuration [17]. To minimise the measurement error related to the fusion splice loss, we used the copropagating pump and signal configuration (Fig. 2). The pump source was a 980-nm single-mode laser diode

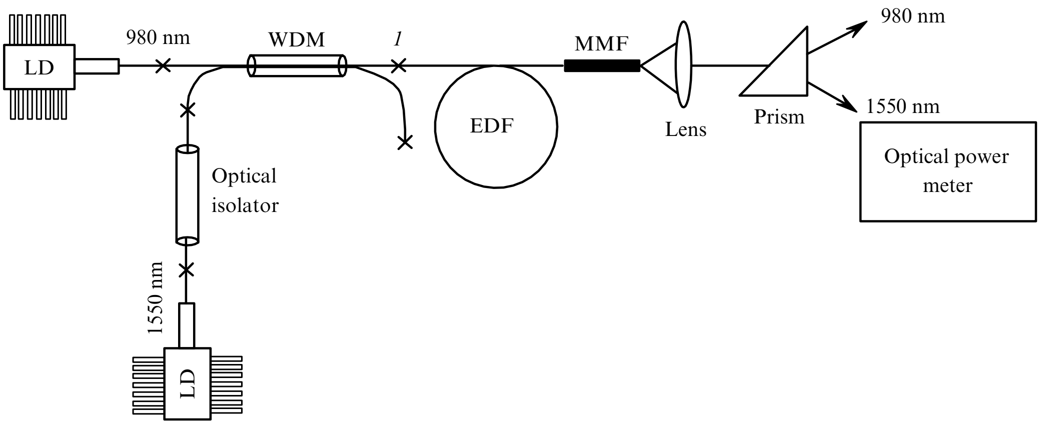

Figure 2. Experimental setup for measurements of the conversion efficiency of erbium-doped fibres.

(LD) with an output power of up to 200 mW. The pump radiation was coupled into the core of the erbium-doped fibre (EDF) using a standard telecom 980/1550-nm wavelength-division multiplexer (WDM). The 10-mW, 1550-nm laser diode signal was also coupled in via this multiplexer. A 1550-nm fiber isolator prevented the fibre amplifier from lasing. The reflection of the output signal from the end face of the erbium-doped fibre was suppressed by fusion-splicing it to a multimode fibre (MMF) with a 60-μm core diameter. The pump and input signal powers were measured directly at the amplifier input: to this end, the active fibre was cutback at point (*1*).

The configuration used allowed us to minimise the experimental errors associated with lasing, spontaneous luminescence and changes in the loss due to fusion splicing of the multiplexer end to the erbium-doped fibres, which differed in core diameter. At the amplifier output, the residual pump light (980 nm) and amplified signal (1550 nm) were separated by a prism and measured separately. The length of the erbium-doped fibre was optimised experimentally so as to maximise the amplified signal power. The pump-to-signal power conversion efficiency (amplifier efficiency) was evaluated as the slope of the plot of $P_{\mathrm{out}} - P_{\mathrm{in}}$ (the difference between the output and input powers at 1550 nm) against pump power coupled into the erbium-doped fibre, P_{p}.

The erbium-doped fibres were divided into several groups according to the core glass composition: phosphosilicate fibres (4 mol % to 10 mol % P_2O_5; P fibres), aluminosilicate fibres (1.5, 3, 6 and 12 mol % Al_2O_3; Al-1.5, Al-3, Al-6 and Al-12, respectively), phosphorus-pentoxide-enriched APS core fibres (~ 4 mol % excess P_2O_5; APS-P) and alumina-enriched APS fibres (1.5, 3 and 6 mol % excess Al_2O_3; APS-Al-1.5, APS-Al-3 and APS-Al-6, respectively). In each group, the erbia concentration was varied widely.

Figure 3 shows the pump-to-signal conversion efficiency as a function of Er_2O_3 content for all the groups of fibres. It is seen that, at high Er_2O_3 concentrations, the highest conversion efficiency is offered by the fibres with aluminosilicate cores containing 3 mol % Al_2O_3 or more. Increasing the Al_2O_3 content of the core from 3 mol % to 12 mol % (fibres Al-3, Al-6 and Al-12) or the addition of $AlPO_4$ (fibres APS-Al-3 and APS-Al-6) causes no increase in conversion efficiency. All the data points fall close to a limiting curve [Fig. 3, curve (*1*)]. The conversion efficiency limit decreases with increasing erbium concentration, probably because the erbium distribution over the glass is nonuniform even when there is no clustering. Accordingly, with increasing erbium concentration there are increasingly more erbium ions that are sufficiently close to one another for energy transfer [2].

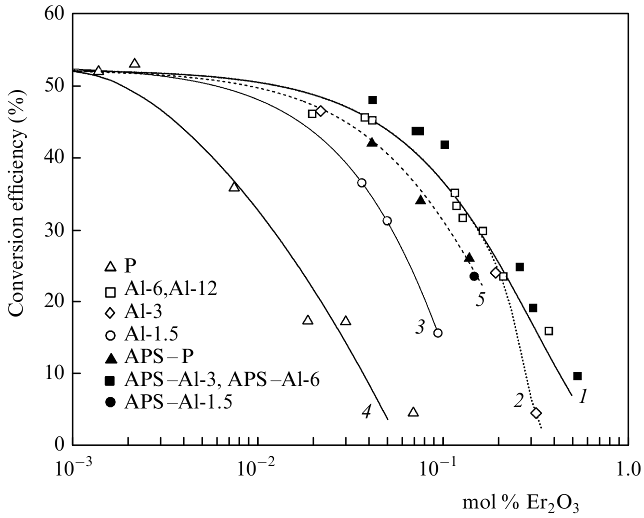

Figure 3. Effect of erbium content and core glass composition on the conversion efficiency of erbium-doped fibres.

In Fig. 3, the conversion efficiency drops to below the limiting curve for the Al-3 fibres containing 0.3 mol % Er_2O_3 [curve (*2*)] and for the Al-1.5 fibres containing more than 0.03 mol % Er_2O_3 [curve (*3*)]. In both instances, the Al_2O_3 concentration is insufficient to prevent erbium clustering, in qualitative agreement with the conclusion drawn by Wagener et al. [1] that at least 20 aluminium atoms per erbium atom are needed.

The erbia solubility in phosphosilicate glasses is much lower than that in aluminosilicate glasses [18]. Accordingly, the conversion efficiency in the phosphosilicate core fibres drops at an order of magnitude lower erbia concentration [Fig. 3, curve (*4*)] in comparison with the Al_2O_3-rich fibres.

The effect of $AlPO_4$ on erbium solubility can be best demonstrated by adding this compound to a host glass highly susceptible to clustering. In this study, a phosphosilicate glass was used as the host. We compared two samples: a P fibre (4 mol % P_2O_5, 0.7 wt % F) and an APS-P fibre (9 mol % $AlPO_4$). In both fibres, the erbia content was 0.07 mol %. The phosphosilicate fibre was doped with fluorine in order to reduce the refractive index of the core to the level of the APS-P fibre, in which the presence of 9 mol % $AlPO_4$ reduced Δn by approximately 0.001. As a result, the two fibres were identical in Δn (Fig. 4a) and cutoff wavelength, which allowed us to avoid errors associated with differences in guidance properties between the fibres. The basic distinction was that the APS fibre contained 9 mol % $AlPO_4$ and the phosphosilicate fibre contained 0.7 wt % F. Conversion efficiency measurements showed that the erbia concentration in question was too high for the phosphosilicate fibre: it essentially lost the ability to amplify the signal [Fig. 4b, curve (*1*)]. At the same time, the conversion efficiency in the APS fibre was about one order of magnitude higher [Fig. 4b, curve (*2*)].

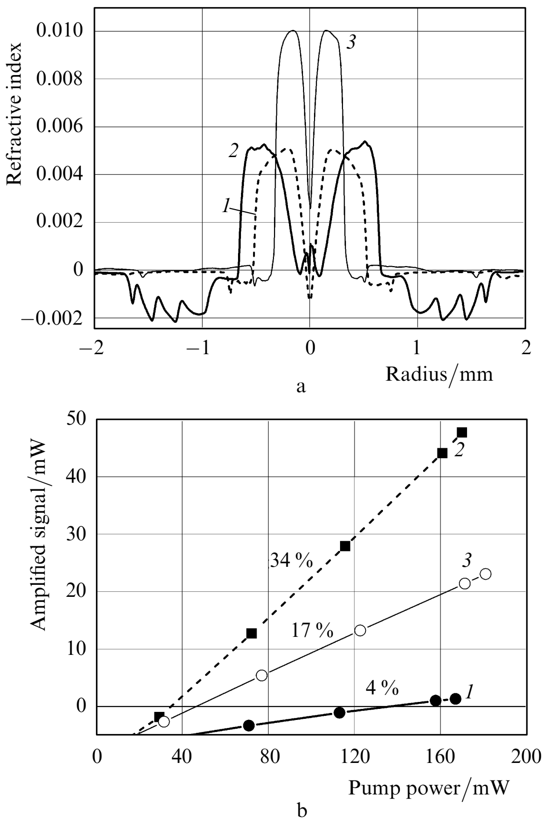

Figure 4. (a) Refractive index profiles and (b) amplified 1.55-μm signal vs. 0.98-μm pump power for erbium-doped fibres with different core glass compositions: (*1*) 6 mol % P_2O_5, 0.7 wt % F; (*2*) 4 mol % P_2O_5, 9 mol % $AlPO_4$; (*3*) 11 mol % P_2O_5.

Comparison with a P fibre that had a factor of 2 higher phosphorus concentration and a factor of 2 lower erbium concentration in the core [Fig. 4b, curve (*3*)] also indicated that the APS-P fibre had better performance. It is worth noting that the P_2O_5 concentration in the phosphosilicate fibre represented by curve (*3*) exceeded not only the excess concentration of P_2O_5 but also its absolute concentration in the APS-P fibre. Moreover, the core of this phosphosilicate fibre was free of fluorine and other dopants for adequacy of comparison. The data in Fig. 4 provides solid evidence that $AlPO_4$ is capable of inhibiting erbium clustering. Comparison of the APS-P and P fibres in a wide range of erbium concentrations [Fig. 3, curves (*4, 5*)] demonstrates that the presence of $AlPO_4$ in phosphosilicate glasses allows the erbia concentration to be raised by almost one order of magnitude with no loss in conversion efficiency.

The data in Fig. 3 demonstrates that the conversion efficiency in the fibres containing more than 3 mol % Al_2O_3 exceeds that in the APS fibres containing excess phosphorus or a slight excess ($\sim$ 1.5 mol %) of Al_2O_3. The aluminosilicate hosts can be doped with erbium to a factor of 1.5 higher concentration, while maintaining the same conversion efficiency. However, because of the high molar refractivity of Al_2O_3, raising its concentration to just 3 mol % increases the refractive index of the core to $\sim 7 \times 10^{-3}$. Even with fluorine doping ($\sim$ 0.7 wt %), the refractive index of the core can be reduced to 0.002 – 0.003 only by lowering the Al_2O_3 content to 1.5 mol %, which will reduce the conversion efficiency in the erbium-doped fibres [Fig. 3, curve (*3*)]. It is reasonable to expect that the use of APS glass would prevent the conversion efficiency from dropping. To verify this assumption, we studied two fibres, Al-1.5 (0.7 wt % F, 0.095 mol % Er_2O_3) and APS-Al-1.5 (30 mol % $AlPO_4$, 0.15 mol % Er_2O_3), having almost identical cutoff wavelengths and refractive indices of the core. The measurement results presented in Fig. 5 demonstrate that, in spite of the factor of 1.5 lower erbium concentration, the Al-1.5 fibre [curve (*1*)] is markedly inferior in con-

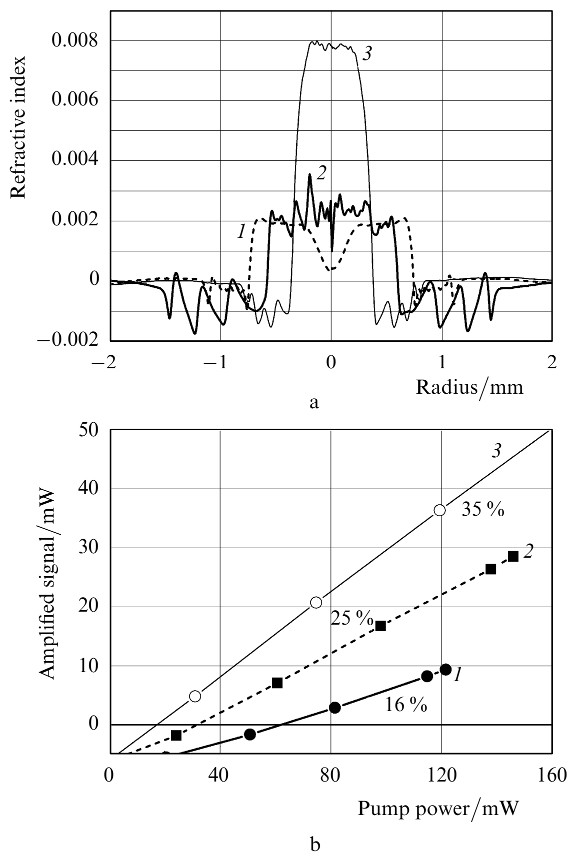

Figure 5. (a) Refractive index profiles and (b) amplified 1.55-μm signal vs. 0.98-μm pump power for erbium-doped fibres with different core glass compositions: (*1*) 1.5 mol % Al_2O_3, 0.7 wt % F; (*2*) 1.5 mol % Al_2O_3, 30 mol % $AlPO_4$; (*3*) 3 mol % Al_2O_3.

version efficiency to the APS-Al-1.5 fibre [curve (*2*)]. As mentioned above, raising the aluminium content to 3 mol % [curve (*3*)] ensures a substantial increase in conversion efficiency but is accompanied by a sharp rise in the refractive index of the core.

Note that, in Fig. 3, the data point for the APS-Al-1.5 fibre lies on the curve for the APS-P fibres [curve (*5*)]. In both instances, the excess codopant (P_2O_5 or Al_2O_3) concentration is insufficient for effective erbium dissolution and, hence, curve (*5*) illustrates the influence of $AlPO_4$ structural groups on Er_2O_3 solubility.

Thus, in the case of erbium-doped fibres with an increased mode area, it is reasonable to use APS glasses as the core material. On the one hand, $AlPO_4$ reduces the refractive index of the fibre core. On the other, because the $AlPO_4$ concentration in the core is high (10 mol % – 30 mol %), this compound enables a marked increase in conversion efficiency even though it dissolves less erbium than does Al_2O_3.

The use of APS glasses for the fabrication of active fibres offers a number of advantages. Phosphosilicate and aluminosilicate glasses are known to have very low photosensitivity, so fibre laser cavities are commonly produced by fusion-splicing an active fibre to passive germanosilicate fibres with Bragg gratings. The splice loss reduces the efficiency of the fibre laser. In the case of APS fibres, doping with roughly equal amounts of alumina and phosphorus pentoxide reduces the refractive index to below that of silica glass, which allows the index profile in such fibres to be controlled using other dopants. Germanium oxide as a codopant would be expected to considerably enhance the photosensitivity of the fibre, enabling Bragg grating inscription directly into the core of the active fibre.

To verify this idea, we fabricated a fibre doped with Al_2O_3 (1.5 mol %), $AlPO_4$ (26 mol %), GeO_2 (5 mol %) and Er_2O_3 (1.5 mol %). Its photosensitivity was compared to that of a germanosilicate fibre containing 4.5 mol % GeO_2 ($\Delta n = 0.007$). To enhance their photosensitivity, the fibres were exposed to a H_2 atmosphere at 100 atm and 100 °C in order to load the glass network with molecular hydrogen. Photoinduced index changes were assessed from the reflectivity of a Bragg grating UV written into the fibres by a frequency-doubled Ar^+ laser (244 nm) using two-beam interference. The rate of the increase in the refractive index of the erbium-doped APS fibre containing GeO_2 [Fig. 6, curve (*1*)] was comparable to that for the germanosilicate fibre, with roughly the same GeO_2 content [Fig. 6, curve (*2*)]. An erbium-doped fibre laser with a cavity formed by two Bragg gratings with 1580-nm reflectivities of 100 % and 10 %, inscribed into the former fibre had a slope efficiency of 43 %.

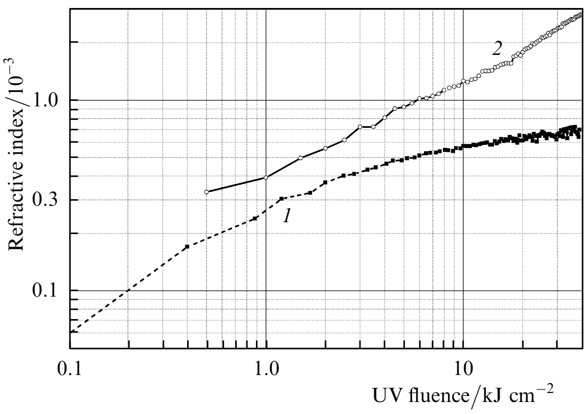

Figure 6. Photoinduced refractive index change as a function of UV fluence for (*1*) an APS fibre doped with 5 mol % GeO_2 and (*2*) a germanosilicate fibre containing 4.5 mol % GeO_2.

4. Conclusions

We have studied the optical properties of erbium-doped APS core fibres in wide ranges of erbia, alumina and phosphorus pentoxide concentrations. The absorption and luminescence spectra of the P_2O_5- or Al_2O_3-enriched erbium-doped APS core fibres are shown to be similar to those of the erbium-doped fibres singly doped with phosphorus pentoxide or alumina, respectively.

The addition of $AlPO_4$ to phosphosilicate glasses enables the erbium concentration to be raised by almost one order of magnitude with no loss in conversion efficiency. The addition of $AlPO_4$ to aluminosilicate fibres with a low Al_2O_3 concentration (1.5 mol %) considerably increases the conversion efficiency at high Er_2O_3 concentrations. Bragg gratings can be inscribed directly into the core of active APS fibres doped with germanium oxide.

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