

MECHANISMS OF OPTICAL LOSSES IN POLYCRYSTALLINE FIBERS

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ABSTRACT

Different mechanisms of extrinsic losses, including aging, in extruded mixed $\text{AgCl}_x\text{Br}_{1-x}$ fibers are discussed. Additional losses caused by changes in structure of fiber material have been investigated.

INTRODUCTION

Mechanisms of optical losses in polycrystalline fibers are still not clear due to their complicated dependence on a lot of various parameters of extrusion process and initial characteristics of crystalline preforms [1-4]. Aging effect is also determined by parameters of "fresh" fibers and by influence of environment conditions and way of fiber use.

Our last studies were undertaken for better understanding of correlation between structure and optical losses in polycrystalline fibers extruded from crystals of solid solutions AgCl-AgBr .

1. FIBER STRUCTURE

The previous structure investigations of polycrystalline fibers were made by light microscopy [5,7], electron microscopy [6,7] and X-ray examination [8]. The optical microscope picture shows the macroscopic structure of fibers, i.e., grain structure and S-shape curly structure (Fig. 4) and [9]. Core/clad fiber structure was examined with optical microscope and will be discussed in forth section.

The average grain size $10 \mu\text{m}$ was revealed by previous electron microscopy measurements [6,7], but the order of magnitude of grain size was estimated $10 \text{ nm} - 100 \text{ nm}$ from a X-raying measurement [8]. Due to this difference in grain size estimation TEM (transmission electron microscope) and SEM (scanning electron microscope) analysis of $\text{AgBr}_{0.75}\text{AgCl}_{0.25}$ fibers was examined for more clear understanding.

The 50 nm width transversal cross-section slices of fiber was prepared by microtom Ultratom-III LKB. The electron microscope "Hitachi" H-500 for TEM analysis was used. Different samples of 9 month old fiber was under investigation.

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Fig.1(a) shows the usual TEM micrograph of fiber cross-section. It is clearly seen the black particles on polycrystalline background. Electrons probe microzond analysis is revealed that black seeing inclusions consist from AgBr mainly. The results of Electron microdiffraction of AgBr particles (with the help of the same "Hitachi" microscope) can be seen on Fig.1 (b); this picture shows the single crystalline structure of this AgBr precipitation. Precipitation of AgBr phase leads to the growth of scattering losses in fiber and is similar to typical phase separation in solid solutions or glasses. Processes of phase precipitation could be one of the mechanisms of aging effect in MIR-fibers along with possible mechanisms of changes in fiber structure with time.

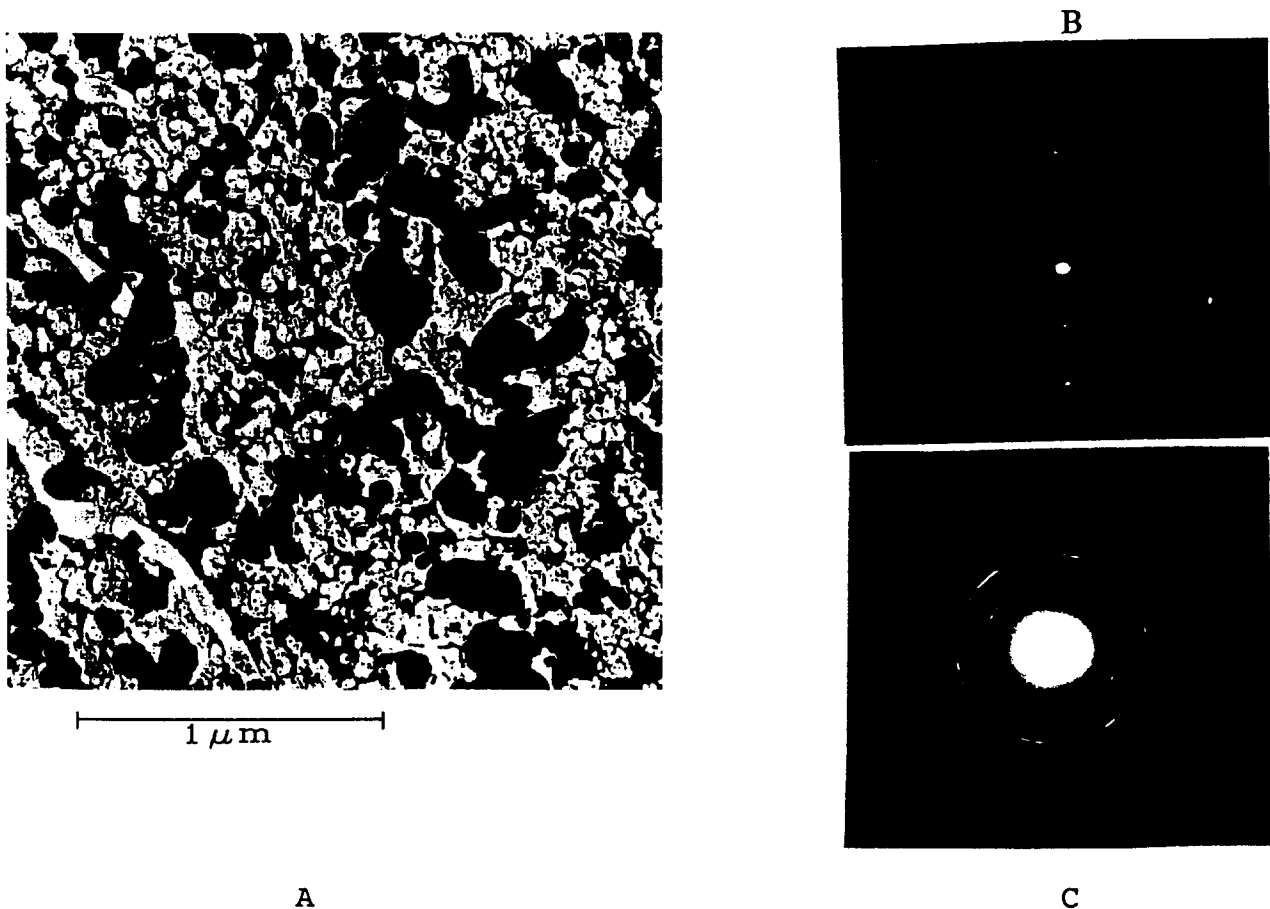


Fig. 1. (A) TEM micrograph of 50 nm fiber slice.
 (B) Electron diffraction pattern of AgBr particles.
 (C) Electron diffraction pattern of polycrystalline structure.

Fig.1(a) also shows polycrystalline structure of the host; the sizes of grains are over the range from 20 nm till 200 nm. The electron diffraction in polycrystalline area was also made by microscope and Fig.1(c) shows the result of that measurements. This measurement confirms the existence of polycrystalline structure. The average size of grains can be calculated from the width of diffraction ring. The order of magnitude of grain size on micrographs is the same as that was measured by Electron microdiffraction and the previous X-raying examination [8].

The intensity of diffraction ring (Fig.1(c)) has non-uniform distribution and it is possible to conclude that fiber has a texture structure with the texture axis 110. Difference between our data and predecessor's measurement [6,7] could be connected with the different extrusion parameters. Structure dependence on extrusion parameters is under investigation now.

Structure of fiber lateral surface was examined by SEM with JEOL-840 electron microscope. Fig.2 presents the view of lateral surface of one month old fiber with fine grain structure on the scale $0.1 \mu\text{m}$. Fig.2 (b) shows the scratch produced by die surface roughness. All that imperfections cause additional optical losses.

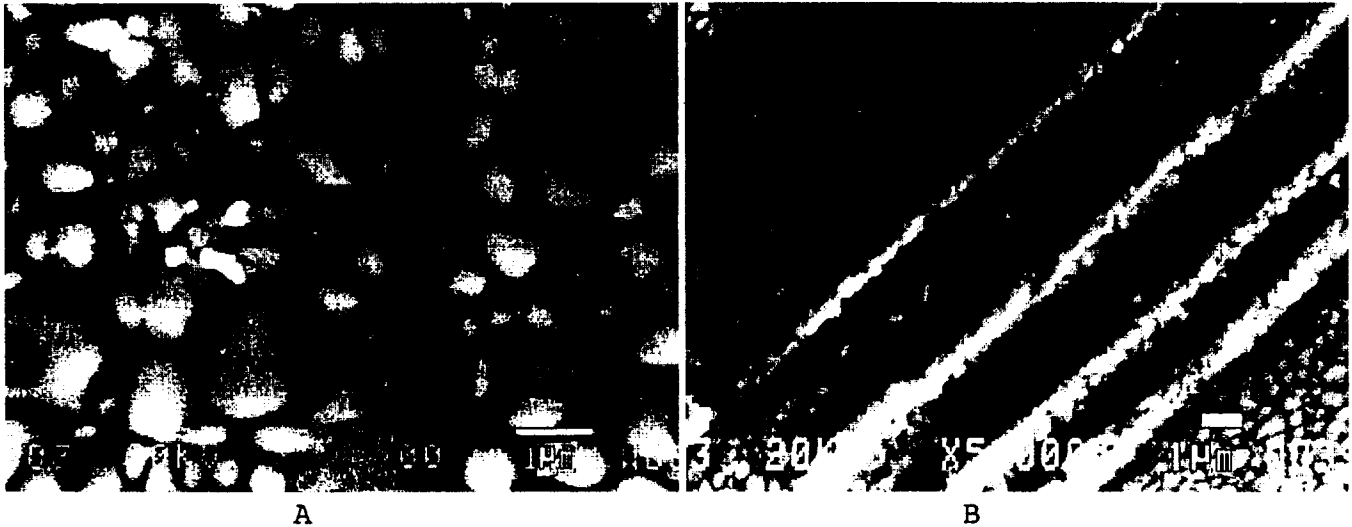


Fig. 2 Electron microscope micrograph.
(A) SEM micrograph of fine grain structure.
(B) The same as (A).

2. OPTICAL FIBER AGING

Process of fiber aging is discussed in this section. Several series of fibers, made by different extrusion methods, were tested during a year (Fig.3).

Attenuation spectra were measured with FTIR - spectrophotometer "Bruker - IFS 113V" in $3-13 \mu\text{m}$. To equalize the mode distribution in fiber we used 15 meter's length starting samples and we cut them at 1.5 mm length in cut back method. Fibers were kept in non-hermetic loose polymer tube for one year at typical laboratory environment condition.

The aging effect is the result of different mechanisms such as structural changes (i. e. phase separation of fiber material), interaction of fiber with atmosphere, external irradiation, which could cause light-induced silver colloid's formation. The contribution of each mechanism is determined by combination of parameters of crystalline preforms, extrusion parameters ambient conditions for storage and manner of fiber use. Now our extrusion method allows to get fibers with total optical losses about $0.6-0.8 \text{ dB/m}$ at $5-6 \mu\text{m}$ and $0.2-0.4 \text{ dB/m}$ at $10.6 \mu\text{m}$, but aging process can develop by different ways (Fig. 3).

In our previous work [8] the set of optical loss spectra was published for fibers with substantial aging effect (Fig. 3(a)). The first spectrum was measured immediately after fiber fabrication. The second one was measured in 5 months after extrusion, the third - in 9 months and the fourth - in 12 months after extrusion. Spectra 3 and 4 are practically identical. Probably, the period about 9 months is enough to stabilize optical properties of silver halide fibers for this kind of aging.

Table 1. Results of fitting.

Number of curve (Fig. 3A)	A, db/m	B	C
1	0.4	39	2.2
2	0.97	115	2.9
3 and 4	1.9	710	3.9

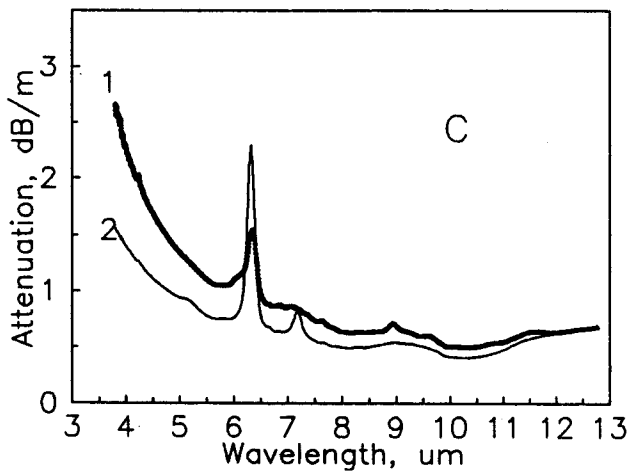
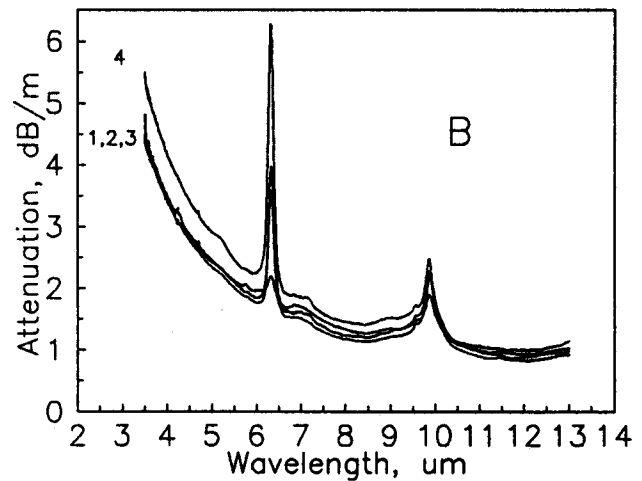
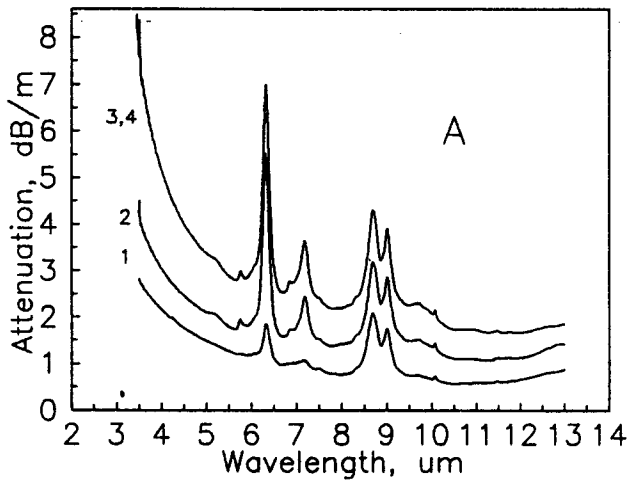


Figure 3. Attenuation spectra of silver halide fibers.

A - the first kind of aging process

1 - immediately after extrusion

2 - in 5 months after extrusion

3 - in 9 months after extrusion

4 - in 12 months after extrusion

B - the second kind of aging process

1 - immediately after extrusion

2 - in 3 months after extrusion

3 - in 6 months after extrusion

4 - in 9 months after extrusion

C - the third kind of aging process

1 - immediately after extrusion

2 - in 3 months after extrusion

TEM slice micrograph of that fiber was made and the view was the same as on Fig.1(a): the AgBr precipitation was born in the polycrystalline host.

All this experimental spectrum was fitted to the function $A+B \cdot \lambda^{-C}$ (range from 3 μm till 5 μm was used for fitting). Results of that procedure are shown in table 1. Degree of power law function was changed after 6 months and become close to 4. This type of loss spectrum's law could be connected with the Rayleigh scattering and the precipitation of small AgBr particles could cause those changes in spectra's law. Rise of constant A is the result of impurity absorption and scattering of optically large particles. The rising value of B can be connected with concentration growth of scattering centers.

Aging effect in KRS-5 fibers was investigated in details by J.A. Wysocki et. al. [10]. Growth of scattering losses was mainly determined by grain boundary separation [2], which may accuse in silver halide fibers also.

The absorption of water at lateral surface of fiber leads to the growth of water absorption peak at 6.2 μm and other correspondent wavelength. We'll not concern so important mechanism as different impurity absorption at all in this paper, because they are still under investigation.

UV- induced growth of absorption in wide spectral region was studied to determine the connection between spectral curve of induced losses and silver colloids formation by several authors [3,11], and for our experiments we just were protecting the fiber samples against UV-irradiation to prevent the noticeable contribution this mechanism.

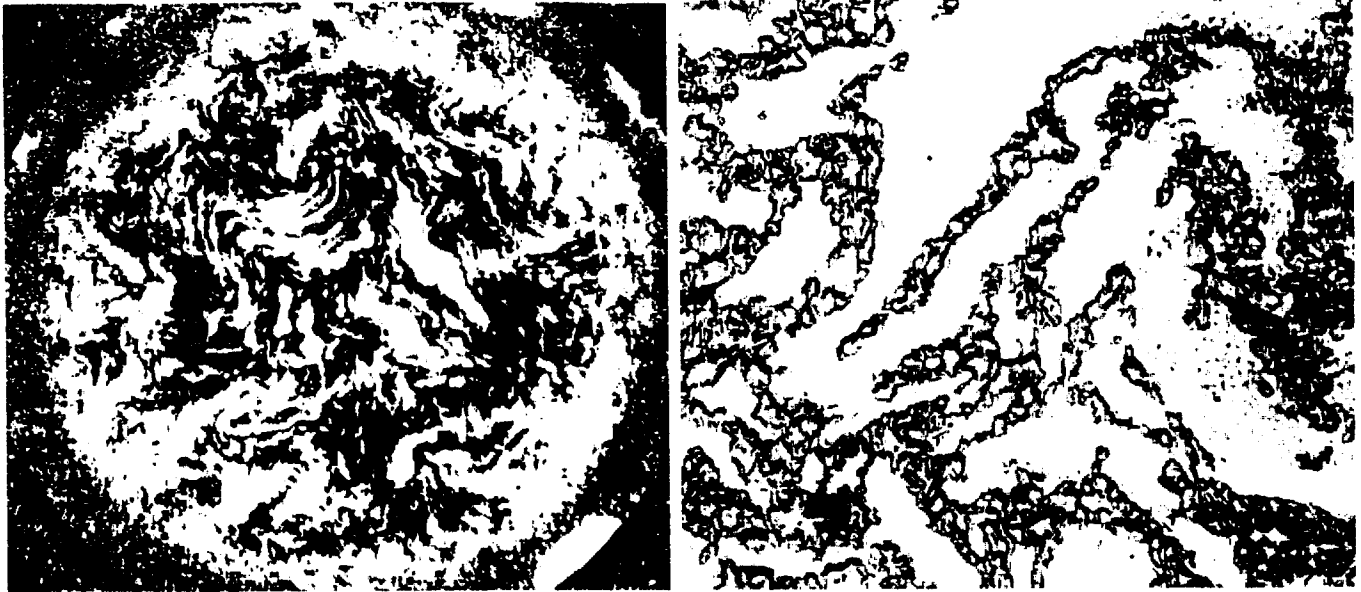
The second sample of fiber (Fig. 3b) represents the behavior of fibers fabricated by modified method. Practically, spectral dependence is the same during 9 months of fiber storage.

The Figure 3(c) shows the spectra of fibers, made by the third version of extrusion method. First results show, that optical losses decrease during 3 month's storage. Probably it is connected with room temperature annealing, which could reduce the scattering losses determined by smoothing of residual stresses and favorable changes in fiber structure. Similar effect of scattering loss redaction was obtained in KRS-5 fibers by annealing [12].

3. CORE/CLAD FIBERS

Spectra of optical losses in core/clad fibers which were extruded by two different methods are shown at Fig.5.

Quality of core/clad boundary for samples with spectrum No.2,3 looks smooth, with roughness much less than 1 μm , but chemical etching reveals the existence of large curled regions in central part of core structure. The view of this structure is known for superplasticity mechanism, where the lamels of one structure phase is moving much easier in less viscous other phase. In our fiber sample polycrystalline lamels with 20-100 μm size (Fig.4a) consisted of 2-5 μm size grains (Fig.4b) and these curved lamels were immersed into another phase of structure, looking like amorphous media due to its super fine structure. Size of structure elements in "amorphous" phase could be close to 10-100 nm, as was confirmed by TEM- microscopy and X-ray examination. Lamels possess of elongated form along the axis of fiber, and it was observed that crystallographic orientation of texture $\langle 110 \rangle$ lies parallel fiber axis. Formation of S-shape



A

B

Fig.4 Optical microscope reflection micrographs of fiber cross-section.

- (A) Transversal cross section after the extrusion of core/clad fiber. The S-shape curly structure can be seen.
- (B) Magnified micrograph of the curly structure of fiber.

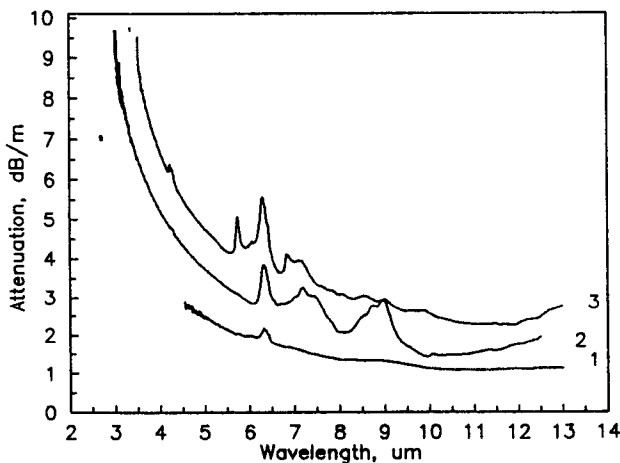


Fig5 Spectra loss of core/clad fibers.
 1 - core-clad fiber spectrum produced by the first method
 2, 3 - core-clad fiber spectra produced by the second method (typical cross-section is shown at Fig. 4a)

curved structure is determined by turbulent flow in extrusion and depends on geometry viscosity and friction parameters along, with substantial role of temperature, speed and pressure of process itself and starting characteristic of crystalline preform.

Non-laminar character of extrusion flow leads to irregularities in core/clad boundary surface, but it seems that for fibers with shown structure it's not a main contributor to total losses. Illustration of this fact is shown at Fig.5. The lowest losses (curve 1) were obtained for fibers with much more rough core/clad boundary, than for fiber No.2 and No.3, where the intersurface was much better (Fig.4 a). The best 3-meter sample of core/clad fiber was obtained with 0.7 dB/m at 10.6 μm .

CONCLUSIONS

In spite of the better understanding of complicated mechanisms of optical losses the basic question of proportions of their contributions in total value is still open. But existed technology of polycrystalline fiber extrusion provides stable fibers with losses less than 0.5 dB/m. To achieve reliable level less than 0.1 dB/m at 10.6 μm the further investigations and technology development is needed, especially for core/clad fibers.

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