Fatigue of mixed silver halide polycrystalline optical fibres

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The transmission of CO₂ laser radiation by polycrystalline $AgCl_xBr_{1-x}$ fibres was measured during cyclic bending of the fibres. These fibres are useful as flexible infrared optical fibres, and it is important to know their fatigue strain limit for applications which require many bending cycles. The investigation was performed in the regime of forced bending vibration. The fibres were found to transmit without significant deterioration after more than 10⁷ cycles. Fatigue strain limit values, based on 10⁷ bending cycles, are in the range of 0.05%–0.30%. These values were found to depend on the composition and to exhibit a maximum for the AgCl_{0.5}Br_{0.5} composition. This dependence on composition has been explained by a theoretical model.

1. Introduction

Silver halide materials have been prepared in our laboratory for the past few years. Mixed compositions of $AgCl_x Br_{1-x}(0 < x < 1)$ are grown from the melt as large crystal ingots. The crystals are then extruded to form fibres in lengths of several metres and diameters of around 1 mm. During extrusion, the crystals are plastically deformed, and the resulting fibres consist of polycrystalline solid-solution materials.

Details of the fibre properties have already been published [1, 2]. They are flexible, highly transparent in the mid-infrared spectral regime of $2-20 \,\mu\text{m}$, nontoxic, very slightly soluble in water, and have an acceptably low sensitivity to ultraviolet and visible light. Various applications of these fibres have been investigated and demonstrated, including infrared spectroscopy [3], radiometry [4], and high-power transmission [5], especially of the CO₂ laser radiation for medical and industrial applications.

The mechanical properties of our fibres, and in particular the hardness and axial strength, have been reported [1]. Axial tension can be avoided by using a suitable cable design, but the ability to bend the fibre should not be excluded, because it is one of the main merits of flexible optical fibres. Measurements of multiple bending properties of the fibres in the plastic bending regime, using a CO_2 laser (at the 10.6 µm wavelength) have already been reported [6]. The values of the elastic strain limit and their dependence on the composition were reported [7].

The practical use of silver-halide optical fibres requires knowledge of their fatigue limit, which we define as the maximum periodic strain which the fibres can withstand for an "unlimited" (> 10^7) number of bending cycles. The multiple bending properties of various infrared fibres in the elastic regime have been investigated [8, 9], but the number of bending cycles in these works was limited to about 60 000. We have already reported [10] the fatigue limit values for AgCl_{0.5}Br_{0.5} fibres. In this work, the fatigue limit of polycrystalline AgCl_xBr_{1-x} fibres (0 < x < 1) was investigated and the dependence on composition was explained by a theoretical model which is based on solid-solution hardening and involves the material constants.

2. Theoretical background

Cyclic strain-controlled fatigue investigation of the fibres is performed in the regime of forced bending vibration. Each symmetric cycle consists of bending of the fibre first in one direction (up) and then in another (down). In bending procedures (as opposed to axial straining) the strain is not uniform in the cross-section of the fibre. The surface of the fibre undergoes maximum strain range and it can be expressed as $\Delta \varepsilon = 2r/\rho$, where r is the radius of the fibre and ρ is the bending radius. Our measurements were carried out for the clamped-hinged boundary conditions, using an apparatus such as that shown in Fig. 1, where the fibre was clamped at the fixed holder and hinged at the moving holder. In this configuration a bending radius, $\rho(x)$, of the fibre changed along its length according to [10]

$$\rho(x) = \frac{L^3}{3h(L-x)} \tag{1a}$$

$$\rho(0) = \frac{L^2}{3h} \tag{1b}$$

where x is the coordinate of every point along the fibre with x = 0 at the clamped end and x = L at the hinged end, h being the amplitude of the moving holder. Because of the symmetric bending, the maximum



Figure 1 The apparatus used for cyclic bending of fibres.

strain range in the fibres was at the clamped end, on the surface of the fibre, and it could be expressed as $\Delta \varepsilon = 2r/\rho(0)$.

Fatigue results are usually presented using strain-life plots (commonly called S-N plots). These plots show the number of cycles to "failure", N_f , as a function of the strain range, $\Delta \varepsilon$. It has been found empirically [11] that N_f depends on $\Delta \varepsilon$ through the Manson-Coffin relationship

$$\frac{\Delta\varepsilon}{2} = \frac{\Delta\varepsilon_{\rm e}}{2} + \frac{\Delta\varepsilon_{\rm p}}{2} = \frac{\sigma_{\rm f}}{E} (2N_{\rm f})^{\rm b} + \varepsilon_{\rm f} (2N_{\rm f})^{\rm c} \qquad (2)$$

where the total strain range, $\Delta \varepsilon$, is a sum of the elastic strain range, $\Delta \varepsilon_{e}$, and the plastic strain range, $\Delta \varepsilon_{p}$. The coefficients are specific constants of the material: E is the Young's modulus, σ_f is the fatigue strength coefficient, $\varepsilon_{\rm f}$ is the fatigue ductility coefficient, while b and c are the elastic and plastic fatigue exponents, respectively. In the regime of small strain-range values, the elastic term, $\Delta \varepsilon_{e}$, dominates (high-cycle fatigue). In the regime of large strain-range, the plastic term, $\Delta \varepsilon_{p}$, dominates (low-cycle fatigue). Comparing the total strain life plots for strong, tough and ductile fibres, we can optimize their properties for different strain-range applications. Around 10³ cycles, corresponding to a total strain of about 2% (low-cycle fatigue), there appears to be no preferred composition [6]. Because the fatigue limit associated with long cyclic life (> 10^7 cycles) is strongly dependent on tensile strength [11], it follows that the fatigue behaviour should be sensitive to the fibre composition.

3. Experimental procedure

Short fibres of various $AgCl_x Br_{1-x}$ compositions were inserted into a Teflon tube to protect their surface. The measurements were carried out for the clampedhinged boundary conditions, mentioned above (see Fig. 1). The moving holder had a conical hole, so that the fibre length and the direction of the exit end could change while bending (this change of the length was negligible, in comparison with the length of the fibre, so we can consider L = const). The beam of a CO_2 laser (Synard model C48-2) was focused into the fibre and the transmission was measured at a power level of about 1 W at the fibre input. A reflective chopper sent part of the beam to a detector, which was used (after calibration) as a reference for monitoring the input power. A power meter (Ophir model DGX) measured the transmitted power at the fibre exit during vibration. The forced bending vibration was produced by a d.c. motor which moved the holder up and down with an amplitude of 2.0–3.5 mm and a constant frequency of 45 Hz. The distance between the fixed and the moving holders was varied form 2.8–6.5 cm, to obtain different bending radii of the fibre. The transmission and the number of bending cycles were measured until the transmitted power dropped to zero, following a mechanical break. A few measurements were interrupted after about 10^7 cycles (the "fatigue limit"). The laser, detectors and motor were controlled by personal computer. The measurements were performed under normal environmental conditions, at a temperature of about 24 °C and humidity of 60%.

4. Results and discussion

The minimal bending radius, $\rho(0)$, was determined from Equation 1 and the maximal strain, $\Delta \varepsilon/2$, in the fibres was calculated. Fig. 2 summarizes our measurements for 0.9 mm diameter fibres of several AgCl_xBr_{1-x} compositions. It is seen that the "mechanical lifetime" of the fibres has some dependence on composition, and that the AgCl_{0.5}Br_{0.5} composition is better than other compositions.

The range of our experimental results corresponds to the transition strain-range between the low-cycle and the high-cycle fatigue. In this range, both the elastic, $\Delta \varepsilon_{e}$, and the plastic, $\Delta \varepsilon_{p}$, terms contribute to the expression for $\Delta \varepsilon$ in Equation 2. The number of cycles until mechanical failure as a function of strainrange is shown in Fig. 3. The graph combines our results for AgCl_{0.5}Br_{0.5} and AgCl_{0.8}Br_{0.2} fibres with few low-cycle fatigue results [6], for several compositions. Examining the low-cycle results, it was concluded [6] that the mechanical failure does not clearly depend on the composition of the fibre. The solid lines in Figs 2 and 3 represent the fit to Equation 2, taking into account all the experimental points. The dashed lines represent the elastic and plastic terms of Equation 2. The same fitting procedure was carried out for all measured compositions. For AgCl_{0.5}Br_{0.5}, the resulting plastic fatigue exponent is c = -0.46 and the



Figure 2 S–N plots for several $AgCl_xBr_{1-x}$ compositions: (\bullet) $AgCl_{0.5}Br_{0.5}$, (\triangle) $AgCl_{0.3}Br_{0.7}$, (\blacksquare) $AgCl_{0.1}Br_{0.9}$.



Figure 3 Cycles to mechanical failure: \bigvee low-cycle results [6]; (O, \spadesuit) high-cycle results. (——) A theoretical fit to Equation 2, (–––) asymptotic lines represent the $\Delta \varepsilon_p$ and the $\Delta \varepsilon_e$ contributions to Equation 2. (O) AgCl_{0.5} Br_{0.5}, (\spadesuit) AgCl_{0.8} Br_{0.2}.

elastic fatigue exponent is b = -0.004. The fatigue ductility coefficient is $\varepsilon_f = 0.25$ and the fatigue strength coefficient divided by the Young's modulus is $\sigma_f / E = 0.0034$. By substituting [7] E = 23.4 GPa, we obtain that $\sigma_f = 79$ MPa. For other highly mixed compositions, σ_f was comparable to the measured values [1] for the fibre tensile strength, which are about 50-90 MPa depending on composition. For compositions close to pure components ($x \approx 0$ or $x \approx 1$) there was some deviation from the predicted values. We explain this deviation by the limited (10^7) number of bending cycles: for highly mixed compositions most of the bending experiments were performed in the elastic regime, and for softer compositions like AgCl_{0.95}Br_{0.05}, only several measurements were carried out in the elastic regime. These fibres are very flexible and do not break while being bent close to the elastic radius after more than 10⁷ cycles.

The behaviour of repeatedly bent fibres can be explained in the framework of mechanical fatigue. This phenomenon is known for samples which undergo numerous stress reversals. The microscopic plastic deformations which are generated in the sample (even for an elastic macroscopic strain) are not completely reversible when the strain is reversed. Permanent defects are formed, and they grow until fracture. In polycrystalline silver halide fibres, microcracks are expected to nucleate along grain boundaries at the fibre surface due to the stress concentration caused by the interaction between persistent slipbands and grain boundaries [12]. In the elastic stress range the dependence of the maximal strain versus number of cycles to failure has the shape of a continually decreasing curve with no apparent lower stress limit, below which the material could be considered completely "safe" [11]. Consequently, the fatigue limit for such materials has to be defined at some specific cyclic life, usually 10⁷ cycles. We used this number for estimation of fatigue limit values.



Figure 4 The fatigue strain limit of $AgCl_xBr_{1-x}$ fibres as a function of the composition, *x*.

A summary of the fatigue strain limit results, based on the 10^7 cycles criterion, is plotted in Fig. 4 as a function of $AgCl_xBr_{1-x}$ composition. The error bars represent the statistical spread of our measurements. It can be observed that highly mixed compositions have the largest fatigue limit. The solid line on the graph corresponds to a theoretical model [13], which was previously used [7] to explain the dependence of the elastic strain limit of $AgCl_x Br_{1-x}$ fibres on composition. The model takes into account the additional internal stress field in mixed crystals - an outcome of the random distribution of different sizes anions in the solid solution. Dislocations in a mixed crystal require a larger work to move against the additional fluctuating stress field, and a higher external stress is thus needed.

According to the model [1, 13], the fatigue limit is a linear extrapolation between the values for the pure components plus the extra internal stress field term. The dependence of the fatigue limit, F, on the AgCl_xBr_{1-x} composition, can be expressed as

$$F(x) = xF(1) + (1-x)F(0) + Ag(x)x(1-x)$$
(3)

where the function g(x) is given by

$$g(x) = \frac{(C_{11} - C_{12})^2}{(C_{11} + C_{12})} \left[\frac{C_{11}(C_{11} + C_{12} + 2C_{44})}{C_{44}(C_{11} - C_{12})} \right]^{1/2} \\ \times \frac{(\Delta V)^2}{l^6}$$
(4)

 $C_{11}(x)$, $C_{12}(x)$ and $C_{44}(x)$ are the elastic constants, l(x) is the lattice constant of the mixed composition, and $\Delta V = \frac{1}{4}(l_{AgBr}^3 - l_{AgCl}^3)$ is the difference of unit-cell volumes. The composition-dependent values of the elastic and lattice constants of $AgCl_x Br_{1-x}$ were interpolated from measured values [14]. The function g(x) varies slowly and monotonically with composition from 29.1 MPa for AgBr (x = 0) to 39.1 MPa for AgCl(x = 1).

The experimental results shown in Fig. 4 are in agreement with Equation 3. The three free parameters of the fit are F(1) and F(0), the fatigue strain limit



Figure 5 The CO₂-laser transmittance of several $AgCl_{*}Br_{1-*}$ fibres during bending to a similar radius, $\rho(0) = 14$ cm. 1, $AgCl_{0.95}Br_{0.05}$; 2, $AgCl_{0.7}Br_{0.3}$; 3, $AgCl_{0.5}Br_{0.5}$.

expected for AgCl and AgBr fibres, respectively, and *A* is a dimensionless constant which characterizes the magnitude of the additional mixed composition stress field. The values obtained for these parameters are: $F(1) \approx 0.011\%$, $F(0) \approx 0.019\%$, $A \approx 32$.

A practical and useful result is the minimal bending radius, $\rho(0)$, calculated for our typical fibres ($r = 450 \,\mu\text{m}$) and shown on the right vertical axis of Fig. 4. The minimal bending radius depends on the composition. In some applications it may happen that a AgCl_{0.5}Br_{0.5} fibre will work in the fatigue strain limit and undergo more than 10⁷ bending cycles, while for a softer fibre these strains are over its fatigue limit and it cannot undergo an "unlimited" number of bending cycles.

Fig. 5 shows the laser transmittance through fibres of different compositions during bending to the similar radius, $\rho(0) \approx 14$ cm. The optical transmission before bending is roughly 70% and the losses are mainly due to Fresnel reflections at the two ends. For most of our fibres, the CO₂ laser transmission did not decrease until close to their fracture. Microscopic observation of broken fibres revealed several cracks, one of which developed until fracture. There were no additional cracks on the spontaneously broken fibres.

5. Conclusion

We have studied the fatigue strain limit of polycrystalline mixed silver halide optical fibres. The experimental method involved measuring of the CO_2 laser transmittance and determining the number of cycles to failure of these fibres in the regime of forced bending vibration.

A dependence on the $AgCl_xBr_{1-x}$ composition was revealed. The values of fatigue strain limit, based on the 10^7 cycles criterion, were found to change in the range 0.05%-0.30%, where highly mixed compositions are much better than the pure components. The behaviour was explained using a theoretical model of solid-solution strengthening and the extra strain to induce plastic deformation in mixed crystals. The model involves estimated composition-dependent elastic constants of the material. An agreement was found between the experimental results for various compositions and the theoretical expression. The fibres were found to transmit the CO₂ laser radiation without deterioration until close to their fracture. The $AgCl_{0.5}Br_{0.5}$ composition would be most suitable for many practical applications.

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References

- N. BARKAY, A. LEVITE, F. MOSER and A. KATZIR, J. Appl. Phys. 64 (1988) 5256.
- F. MOZER, N. BARKAY, A. LEVITE, E. MARGALIT, I. PAISS, A. SA'AR, I. SCHNITZER, A. ZUR and A. KAT-ZIR, Proc. SPIE 1228 (1990) 128.
- S. SIMHONY, I. SCHNITZER, A. KATZIR and E. M. KO-SOWER, J. Appl. Phys. 64 (1988) 3732.
- 4. A. ZUR and A. KATZIR, Appl. Phys. Lett. 53 (1988) 2474.
- 5. D. GAL and A. KATZIR, *IEEE J. Quant. Electron.* **QE-23** (1987) 1827.
- 6. N. BARKAY and A. KATZIR, Proc. SPIE 1591 (1991) 50.
- 7. Idem, J. Appl. Phys. 74 (1993) 2980.
- M. SATIO, M. TAKIZAWA and M. MIYAGI, J. Lightwave Technol. 6 (1988) 233.
- 9. V. G. ARTJUSHENKO, Proc. SPIE 1228 (1990) 12.
- 10. A. GERMAN, N. BARKAY and A. KATZIR, *Appl. Optics* **33** (1994) 2734.
- 11. R. W. HERTZBERG, "Deformation and fracture mechanics of engineering materials" (Wiley, New York, 1983).
- J. T. FONG and R. G. FIELDS, "Basic Questions in Fatigue," Vol. 1 (American Society for Testing and Materials, Philadelphia, PA, 1988).
- 13. T. KATAOKA and T. YAMADA, *Jpn J. Appl. Phys.* **16** (1977) 1119.
- 14. L. S. CAIN, J. Phys. Chem. Solids 38 (1977) 73.

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