World Academy of Science, Engineering and Technology
International Journal of Materials and Metallurgical Engineering
Vol:7, No:5, 2013

Behavior of Optical Fiber Aged in CTAC Solutions

R. El Abdi, A. D. Rujinski, R. M. Boumbimba, and M. Poulain

***Abstract*—**The evolution of silica optical fiber strength aged in cetyltrimethylammonium chloride solution (CTAC) has been investigated. If the solution containing surfactants presents appreciable changes in physical and chemical properties at the critical micelle concentration (CMC), a non negligible mechanical behavior fiber change is observed for silica fiber aged in cationic surfactants as CTAC which can lead to optical fiber reliability questioning.

The purpose of this work is to study the mechanical behavior of silica coated and naked optical fibers in contact with CTAC solution at different concentrations.

Result analysis proves that the immersion in CTAC drastically decreases the fiber strength and specially near the CMC point. Beyond CMC point, a small increase of fiber strength is analyzed and commented.

***Keywords*—**Optical fiber, CMC point, CTAC surfactant, fiber strength.

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I. Introduction

SURFACTANTS are used as detergents, dispersants or pharmaceutical adjuvants and industry uses enormously the chemical activity of surfactant for example for lubricants of machinery. This relies on the fact that a solution containing surfactants presents high changes at critical micelle concentration (CMC). These changes affect the physical and chemical solution properties such as electrical conductivity, surface tension, and detergent activity [1]–[4]. At the CMC point, the water surface tension is reduced by the surfactant which adsorbs the liquid-gas interface. Above this point, stable aggregates are spontaneously formed.

On the other hand, for cleaning industries, it is economically important to find the CMC point because the detergent activity does not effectively change after this point.

To find the CMC point, different techniques are used as fluorimetry, anisotropy probe, spectrophotometry [5], ion-selective electrode, light scattering, conductometry, fluorescence anisotropy probe, and polarography.

Based on the measurement of evanescence wave adsorption [6]-[12], the optical fiber sensors are use more and more. But near the CMC point, surfactants adsorb at solid/water interfaces (particularly at the surface of hydrophilic oxide of silica fiber) and lead to an important decrease of the mechanical fiber structure as the fiber strength and the polymer coating can be seriously damaged.

Using dynamic tensile test, the evolution of mechanical fiber properties (as fiber strength and fiber damage) versus the surfactant concentration is analyzed. Optical fibers are aged during 7 days in cationic CTAC surfactant at 25°C and their behavior is particularly analyzed near the CMC point.

II. Experimental

A. Surfactant Used

Cetyltrimethylammonium chloride solution (CAS number 112-02-07) is a cationic surfactant used as an antiseptic very toxic especially against aquatic bacteria but can also used as a phase-transfer catalyst under conditions which avoid emulsions. CTAC was purchased from Sigma Aldrich Co. (France) (25 wt. % in H_2O). Table I gives CTAC properties and detailed formula.

TABLE I
Physical and Chemical CTAC Properties

Product	Cetyltrimethylammonium chloride solution (CTAC)
Formula	$C_{19}H_{42}ClN$
Molecular weight (g/mol.)	320
pH at 20°C Concentration: 20g /L	6 - 7
CMC* at 25°C (mm/L)	1.3
Boiling point (°C)	100
Density (g/cm^3)	0.968

B. Fiber Used

The used multimode optical fiber has two acrylate coatings (primary and outer coatings). This fiber has a numerical aperture of 0.2 (NA value). A soft, primary coating has a low module of elasticity, adheres closely to the glass fiber and forms a stable interface. It protects the fragile glass fiber against microbending and attenuation. The outer coating protects the primary coating against mechanical damage and acts as a barrier to lateral forces. It has a high glass temperature and Young modulus. It has a good chemical resistance and serves as a barrier against moisture.

The optical fiber core is made of silica and is hydrophilic. If the acrylate polymer coating protects the fiber from mechanical and chemical damages, stress and water cause microscopic flaws in the glass to propagate resulting in fiber failure. Fig. 1 gives the morphology of broken optical fiber.

R. El Abdi is with Rennes1 University, Larmaur Laboratory. Campus de Beaulieu, CS 74205- 35042 Rennes Cedex, FRANCE (phone: 33 2 23 23 41 12; fax: 33 2 23 23 41 12; e-mail: relabdi@ univ-rennes1.fr).

A. Dumitrache Rujinski is with Universita Politechnica of Bucarest, Splaiul Independentei, 060042 Bucarest, Romania (e-mail: ad_rujinski@yahoo.com)

R. Matadi Boumbimba is with Rennes1 University, Larmaur Laboratory. Campus de Beaulieu, CS 74205- 35042 Rennes Cedex, FRANCE (e-mail: r.matadiboumbimba@i2m.u-bordeaux1.fr).

M. Poulain is with Rennes1 University, Photonic Materials Laboratory. Campus de Beaulieu, CS 74205- 35042 Rennes Cedex, FRANCE.
(e-mail: marcel.poulain@univ-rennes1.fr).

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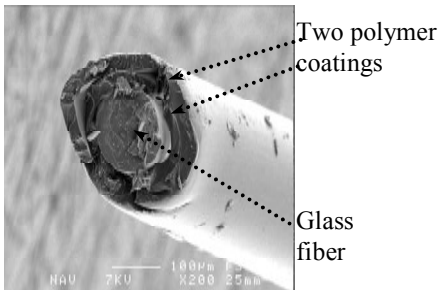


Fig. 1 Morphology of broken silica optical fiber

Optical fibers with and without coatings are analyzed. For naked fibers, the coating was removed on 30mm length (the overall length of tested fiber being equal to 200mm) using an air blower at high temperature.

The naked fibers are very delicate to be tested and cannot be in the contact with impurities such as the dust particles or the manipulator hands. Therefore, only a part of coated fiber was stripped. When the naked fibers are removed from the solution surfactant, they are dried under a drying oven where the air has been filtered to protect the fibers from the environment impurities. As soon as the fibers have been removed from the drying oven, the manipulator (with rubber gloves) performs very fast the tensile tests.

C. Used Test Bench

The dynamic tensile test consists of subjecting fibers to a deformation under a constant velocity until rupture. The fiber is rolled three times around two pulleys (Fig. 2); the lower pulley is fixed and the upper pulley is movable with different velocities (20, 50, 200, and 500mm/min). These strain rates, expressed as a percentage of the initial sample length (200mm), correspond to 1.67 $10^{-3}s^{-1}$, 4.17 $10^{-3}s^{-1}$, 1.67 $10^{-2}s^{-1}$, 4.17 $10^{-2}s^{-1}$.

Tensile testing was performed in a controlled environment with 46-52% relative humidity with a maximum of 5% humidity variation for each series of the tensile tests.

During the test, the deformation and the tensile load are measured using a dynamometric cell while the fiber deformation is deduced from the displacement between the fixed lower pulley and the mobile higher pulley (Fig. 2).

The testing procedure uses 20 samples for each surfactant concentration and for each velocity.

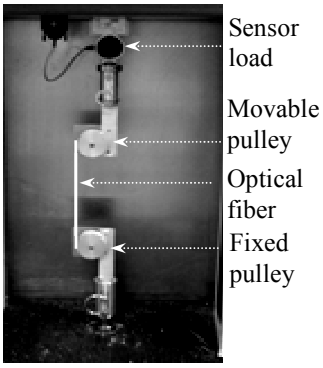


Fig. 2 Dynamic tensile set-up

Before the dynamic tensile tests, optical fibers are plunged in a container with a distilled water- surfactant solution and aged at different surfactant concentrations. This container itself is deposited in water at 25°C. An adiabatic enclosure maintains a constant temperature during the ageing duration of 7 days (Fig. 3).

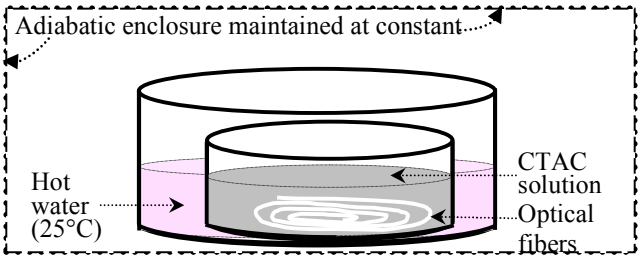


Fig. 3 Fiber aging in CTAC solution

III. Results and Discussion

For coated fibers, Fig. 4 gives the changes of the average failure stress for an aging duration of 7 days in CTAC solution at various concentrations and for different tensile velocities.

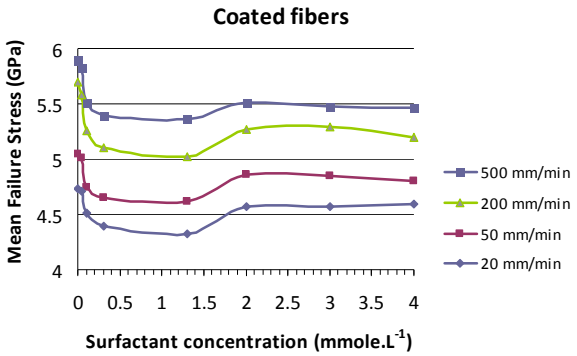


Fig. 4 Mean failure stress change versus CTAC concentration for coated fibers for different tensile velocities

The mean failure stress decreases when the velocity decreases. According to surfactant concentration, the failure stress decreases until reaching a minimum value at CMC critical concentration (1.3mmole/L). Beyond the CMC point, a small failure stress increase is observed. The stress decrease is due to the combined effects of water and surfactant.

A. Water Effect

The silica fiber strength degradation in distilled water is controlled by increasing surface roughness due to the dissolution of silica on the surface of the fiber by water corrosion [13]–[14]. Flaws in glass optical fibers subjected to stress in the presence of moisture grow in a critical way prior to failure. That is due to the combination of stresses at the crack tip and the effect of reactive species, especially water, in the environment.

When a water polar species ruptures the silicon-oxygen bond, dissolution occurs controlled by the following equation:

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$$SiO_2 + H_2O \rightarrow Si-O-H \quad H-O-Si \qquad (1)$$

Silicon-oxygen bonds are slowly broken progressively advancing the crack and the fiber is weakened.

If one can segregate the water effect from the silica surface, the fiber strength cannot present a notable decrease (perhaps a minor decrease can be observed and will be due to the residual moisture inserted between the silica surface and the polymer during the coating application). The fiber strength change after aging depends thus on the permeability of the used coatings, only hermetic coatings are considered capable of completely preventing water from reaching the glass surface [15] and the used acrylate coating shows a small permeability to water diffusion.

B. Adsorption Effect

Surfactant molecules comprise heads and tails. Heads are hydrophilic components and tails are hydrophobic components. These molecules have a component which is water insoluble and another component which is water soluble and thus can diffuse in water and adsorb at interface between water and air. For cationic surfactants as CTAC solutions, hydrophilic part is positively charged and releases a positive charge (cation) in aqueous solution.

At 25°C, the hydrophilic groups of the CTAC molecules dissolve in water before the adsorption onto the silica surface which comprises hydrophilic hydroxide groups OH (1) and onto the hydrophilic polymer coating. When the concentration is below the CMC point, surfactant molecules are scattered in solution, a small adsorption is initiated onto the optical fiber and molecule hydrophobic parts are attracted onto the surface of the interface between the air and the surfactant solution [8].

At the CMC, all the surfaces of optical fiber have been covered with the monolayers of surfactant molecules. Beyond the CMC point, several surfactant molecules, after adsorption onto fiber surfaces, wrap the fiber surface with lasting layers of surfactant aggregates, leading to an increase in fiber thickness, preventing or slowing down the water effect and lead to a weak increase of the fiber strength.

The same behavior is observed for naked fibers but the harmful effect of distilled water and the surfactant leads to severe fiber damage and to a stress decrease higher than for the coated fibers (without coating, fibers are not protected against water and surfactant solution effects). The minimum failure stress value is obtained when the surfactant concentration reaches the CMC point and a small increase is observed beyond CMC concentration (Fig. 5).

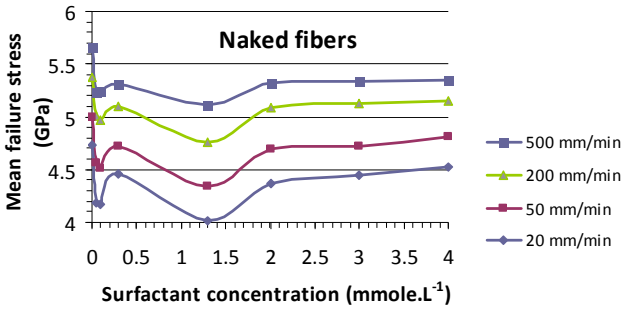


Fig. 5 Mean failure stress change versus CTAC concentration for naked fibers for different tensile velocities

For naked fibers aged in CMC concentration, a high coating damage is observed (Fig. 6). The fiber core is broken and the two polymer coatings are separate after a severe attack from CMC solution. On the polymer external surface (Zoom of Fig. 6), a dense and continuous network of microscopic cracks appears and quickly weakens the fiber resistance.

One observes a disintegrating of the inner coating. The external coating is much deteriorated (several pieces are removed); the polymer is torn with large cracks: the fiber is highly damaged.

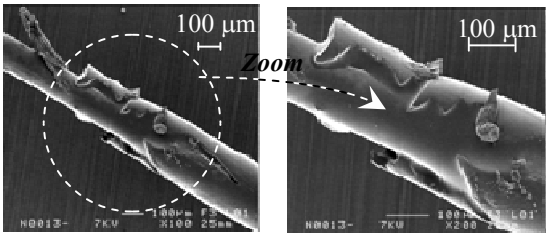


Fig. 6 Fracture morphology for aged naked fibers
Surfactant concentration 1.3mmol./L (CMC)
(Aging period =7 days) (Tensile test velocity of 200mm/min)

IV. Conclusion

Fiber mechanical behavior immersed in Cetyltrimethylammonium chloride solution is analyzed.

The experimental results illustrate the change of the strength curve at the point of the critical micelle forming molecules. The use and the quality of the fiber coating are determining factors for the strength change for fibers immersed in the surfactant solution.

Near the critical CMC point, the CTAC adsorption leads to the formation of a CTAC monolayer on the fiber surface and beyond this point the adsorption amount remained unchanged as the fiber strength. Since the whole surface of the optical fibers is covered with surfactant molecules as soon as the surfactant concentration reaches the CMC, the water effect is reduced, the fiber thickness increases and a small increase of fiber strength is obtained.

The adsorption of CTAC depends on the silica and coating surfaces and the decreasing of fiber strength can be prevented if hermetic coating is used for fiber sensors used in harsh environments as in chemical industries.

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R. El Abdi is a professor at Rennes1 University (Rennes Applied Mechanics Laboratory). He is specialized in the structure behavior analysis, in fracture fatigue, damage and mechanical engineering. His current research interests include analysis of numerical methods, finite-element methods, boundary integrals, nonlinear damage modeling and experimental optic fiber analysis. He manages a mechanical team which is interested in surfaces and contact problems.