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ScienceDirect
Journal of Non-Crystalline Solids 352 (2006) 3556–3560
JOURNAL OF NON-CRYSTALLINE SOLIDS
www.elsevier.com/locate/jnoncrysol

Mechanical properties of optical glass fibers damaged by nanoindentation and water ageing

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Available online 24 July 2006

Abstract

Mechanical properties of optical glass fibers are strongly influenced by cracks on glass surface. Cracks may be generated during manufacture and handling. The chemo-mechanical effect due to water may decrease the response of glass fibers because of surface degradation. In this work glass fibers were aged in a wet chamber at humidity of 85% in temperatures lower than 90 °C, during seven weeks. After being aged, the fibers were submitted to tension tests. The fracture loads were investigated by Weibull statistics. An increase in dynamic fatigue parameter was observed. Surface analysis by AFM indicated smooth surfaces. Small cracks on optical fiber glass surface were made by Vickers indentation and nanoindentation using a Berkovich indenter. A decrease on applied load to fracture indicates that the fiber was damaged by indentation. The influence of indentation load was investigated. As the deformation rate was very low during tension tests, the cracks grow during the test and the fiber fracture at loads lower than for not indented fibers. The effect of water and small indentation fracture on glass is discussed considering the surface damage induced by both methods. The size of the cracks from the different indentations and ageing are estimated.

PACS: 61.43.Fs; 62.20.Mk; 62.20.Qp

Keywords: Optical fibers; Fracture; Indentation, microindentation; Water

1. Introduction

Optical fibers must present high mechanical and corrosion resistance, since they are submitted to chemically aggressive environments during all the time. The effect of corrosion decreases the mechanical resistance of fibers [1]. The decrease in mechanical resistance promotes rupture in mechanical loads lower than the typical values obtained during tests in pristine fibers. The ageing effects may be investigated in order to understand how the fibers can degrade in aggressive environments.

Water is one of the most aggressive agents that degrade silica optical fibers since the surface of silica glass is attacked by water as verified by Michalske and Freiman [2]. The crack grows as the silicon–oxygen bondings are broken by OH^{-1} radicals from water molecules. Other corrosion process may be avoided, but water is always present in the environment. The study of this effect is very important. The polymer is used to protect the glass from external environment. However, this protection is not perfect since the water can diffuse into the polymers and reach the glass surface.

The mechanical behavior of optical fibers may be determined by static fatigue. In this case, a constant load is applied and the rupture is observed after some time, if the load is higher than a threshold load is applied. The fracture depends on the load, the quality of the coatings,

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doi:10.1016/j.jnoncrysol.2006.02.114

and the temperature, but mainly the presence of water in the environment. Dynamic fatigue is another used test that consists of a constant loading rate applied to the fiber up to rupture.

Fibers may also age without any applied load. In this case, the mechanical properties vary with time due to chemical attack from a corrosive environment. The modification in the mechanical resistance of the fibers with time is called as zero load ageing.

Small particles and other fabrication defects can decrease the mechanical resistance of the fibers. Damage fibers can be removed by the proof test made during manufacturing at a constant stress of 0.7 GPa [3].

The effects of flaws in fibers were investigated by Lin et al. [4] and Semjonov et al. [3]. They analyzed the effect of indentation on glass surface on the mechanical resistance of the fibers. The decrease in rupture loads during bending and tensile tests were investigated as a function of the indentation load. Lin made indentations with Vickers indenters and high loads. Results from cube corner indentations and lower loads were reported by Semjonov.

An important parameter to describe the quality of an optical fiber is the fatigue susceptibility '*n*' given by the equation [5]

$$\log(\sigma_f) = \frac{\log(\dot{\sigma})}{(1+n)} + c, \tag{1}$$

where σ_f is the fracture stress and $\dot{\sigma}$ is the stress rate and c is constant. The parameter n is directly related to the mean velocity of crack growth. Then, under the presence of water the crack velocity is higher and n is lower. In fibers optics, n is commonly related to the humidity (radical OH) present in the fiber surface [5]. High quality fibers present high values of n since this parameter is related to the mean velocity of crack growth.

In this work the mechanical properties of silica glass optical fibers aged for seven weeks in humid environment were investigated by tensile tests. Indentations and scratch tests were performed with Vickers, Berkovich and cube corner indenters to create controlled flaws into the glass surface of the fibers. The effect of these controlled flaws was investigated considering the fiber resistance during tensile tests.

2. Experimental procedure

The commercial silica glass optical fibers tested in this work were produced by vapor diffusion apparatus (VDA) (fiber A) and outside vapor deposition (OVD) process (fiber B). The complete fibers have 250 μm of diameter while the glass diameter was 125 μm.

Ageing of fiber B was made in a chamber at temperatures of 85 °C and humidity of 85% during seven weeks. Every week, a part of the fibers was removed from the chamber and submitted to a tensile test.

The tensile tests were made in Testware II model 810 equipment from MTS at Universidade Tecnológica Federal do Paraná (UTFPR) with a load cell of 100 N. The displacement rates were 0.5, 5, 50 and 500 mm/min. For each rate 15 fibers were tested.

Polymer coatings over glass cladding were removed by sulfur acid at 200 °C during 10 s, in order to perform the indentations and scratches in the glass surface.

Vickers indentations were made with a Shimadzu durometer. The applied loads were 100 mN. The indenter was oriented in such a way as to have one diagonal parallel to the fiber axis. Berkovich indentation tests were made in the Nanoindenter XP from MTS at loads of 10 mN. The scratch tests were made with Berkovich and cube corner indenters at loads of 1 mN and 10 mN. Channels in a V format, with depths of 125 μm, were made in an aluminum rod to support the fibers to perform scratches and indentations. The scratch paths were perpendicular to the fiber axis with the indenters oriented with an edge in the scratch direction. The total scratch length was about 1 mm starting from the metal support, crossing the fiber and finishing in the metal support.

The tests were made at temperatures of 23 ± 3 °C at an air humidity varying from 50% to 80%.

3. Results

3.1. Ageing effects

The probability of fracture in the tensile test for the pristine and the four weeks aged fiber type B is shown in Fig. 1, for the 5 mm/min rate test. It is possible to verify a decrease in fiber resistance (σ) from about 4.1 GPa to about 2.9 GPa. The parameter m of Weibull statistics decrease from 51 for pristine fiber to 31 for the aged fiber. This parameter m is related to the variability of the mechanical resistance. High values of m indicate closer values while low m indicates large dispersion. Pristine fibers have a high

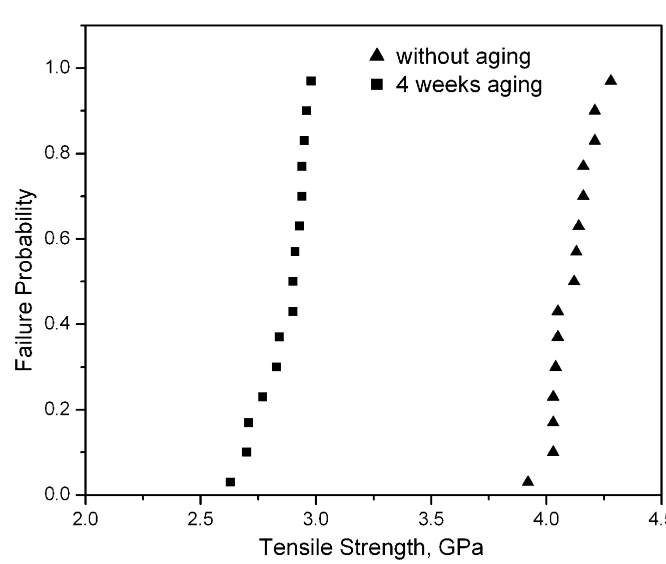

Fig. 1. Failure probability of fiber B aged for four weeks and for the pristine fiber. The tensile test deformation rate was 5 mm/min.