Strength and surface characterization of aluminum-coated fused silica fibers

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ABSTRACT

Hermetic aluminum-coated fused silica fibers can withstand high stress levels without failure for prolonged periods of time in water-containing environments. Aluminum-coated fibers from several sources exhibit differences in strength. The aluminum and silica surfaces have been examined using SEM and AFM in order to understand this variation. Differences in the interfacial interaction between aluminum and glass and in the microstructure of the coatings were considered, but were not unequivocally identified as being responsible for the differences in strength observed for the various aluminum-coated specimens.

Keywords: reliability, strength, hermetic, optical fibers, aluminum-coated fibers, AFM.

1. INTRODUCTION

Mechanical reliability of optical fibers is as important as their optical properties for practical applications. The ambient environment is known to degrade the strength of optical fibers even in the absence of any applied stress.[1,2] Coatings can protect fibers from damage caused by exposure to the environment. Polymer coatings do provide protection from mechanical damage, but are permeable to water and so do not stop water reacting with the glass surface. Therefore, hermetic coatings have been developed which exclude water from the fiber surface. Many materials have been developed for making hermetic coatings, both non-metallic, such as carbon, SiC, TiN, SiON, and metallic, such as Al, Sn and Zn.[1,3] Inorganic hermetic coatings may themselves be more brittle than the glass fibers and may only be effective up to a relatively low stress. In contrast, metal coatings are found to improve resistance to fatigue while still maintaining high strength.[4]

Aluminum is a material which has been studied as a coating for silica fibers since 1964[5] and for optical fibers since 1979.[6] However, no aluminum coated fibers were made that were substantially stronger than polymer coated fibers until 1988.[7] It is not clear why other aluminum coated fibers are so much weaker than the strengths reported by Bogatyrjov, *et al.*.[7] The purpose of this work is to investigate the surface and interfacial properties of five fibers with different strengths obtained from five different manufactures, in order to understand the source of the strength variability.

2. EXPERIMENTAL PROCEDURE

All five fibers used in this study were coated with aluminum using the freezing method[8] in which a fiber is passed through an orifice surrounded by molten aluminum. The fiber entrains some molten aluminum which

Table 1. Fiber and coating diameters of aluminum-coated fibers from five manufactures.

Fiber	A	B	C	D	E
Fiber Diameter (μm)	175	125	125	125	125
Coating Diameter (μm)	215	170	165	175	160
Coating Thickness(μm)	20	22.5	20	25	17.5

Table 2. Mean Strain to failure and Weibull modulus of aluminum-coated fibers.

Fiber	A	B	C	D	E
RT, coated, failure strain	9% 8%	14%	11.8%	11.4%	12%
Weibull modulus, *m*	10-20 3	120	15	—	25
LN_2, failure strain	11.4% (coated)	17.8% (bare)	—	—	—
RT, stripped, failure strain	5%	8%	—	8%	—

rapidly freezes leaving a smooth, thin metal coating on the fiber surface. The coating diameters range from 160 to 215 μm, while the glass diameter is 125 μm for all specimens except fiber A which has a 175 μm glass diameter (Table 1). A dynamic two-point bending technique[9] was used to determine the strength of these five fibers. Fibers were tested in ambient laboratory air (~25°C) as well as in liquid nitrogen, both with and without coatings. However, not all fibers were tested in all environments (Table 2). For measurements on bare fibers, the aluminum coating was removed by dipping the fiber in 40°C $FeCl_3$ solution which has been shown to have no effect on the strength of uncoated silica fibers. A Nanoscope II† atomic force microscope (AFM) was used to image the bare fiber surfaces. The microstructure of the aluminum coatings were studied by scanning electron microscopy (SEM) using an Amray 1200C.‡

3. RESULTS AND DISCUSSION

The strengths of the fibers are shown in Table 2. They are given as the mean strain to failure; the failure stress is not used since bending directly measures failure strain, and the elastic modulus is not known at these high strains. In all environments, fiber B is the strongest, fiber A is the weakest, while the other fibers have intermediate strengths. Since the coating application process was similar for all fibers, it is important to understand what causes the strength differences in order to be able to fabricate the strongest fiber possible.

Fiber B tested stripped and in liquid nitrogen (LN_2) gave 17.8% failure strain. This value is close to the pristine strength[10] indicating that the aluminum coating has not damaged the glass. The coated fiber strength at room temperature is somewhat lower. This suggests that the coating traps some moisture at the glass surface which results in some fatigue at room temperature. In contrast to fiber B, fiber A has substantially lower strengths and Weibull moduli under all conditions. Two sets of coated room temperature measurements for fiber A are shown in Table 2; they were obtained from different specimens. While they show similar mean strengths, there is a substantial difference in the Weibull moduli, indicating batch differences. We also observed considerable variability in the mean and deviation of strength along the length of a single fiber, suggesting substantial process variability for this specimen. It is clear that fiber A has suffered irreversible strength degradation upon coating since its broad and weak strength distribution does not recover to a narrow pristine strength distribution upon stripping. These results show that the aluminum coating can influence the fiber strength either considerably and permanently or only slightly.

One possible cause of the strength degradation of aluminum-coated fibers is the interfacial reaction between aluminum and silica glass which might enlarge the surface flaws[11] or roughen the fiber surface. Arridge and Heywood[5] suggested that this might be the reason for the scatter in their strength results. Bogatyrjov *et al.*[12] showed that on heat treating aluminum coated fibers, the strength degradation which occurred could be correlated with the size and growth of crystals of alumina seen by others.[13] The activation energies for nucleation and

† Digital Instruments, Inc., Santa Barbara, California, USA.
‡ Amray Inc., Bedford, Massachusetts, USA.

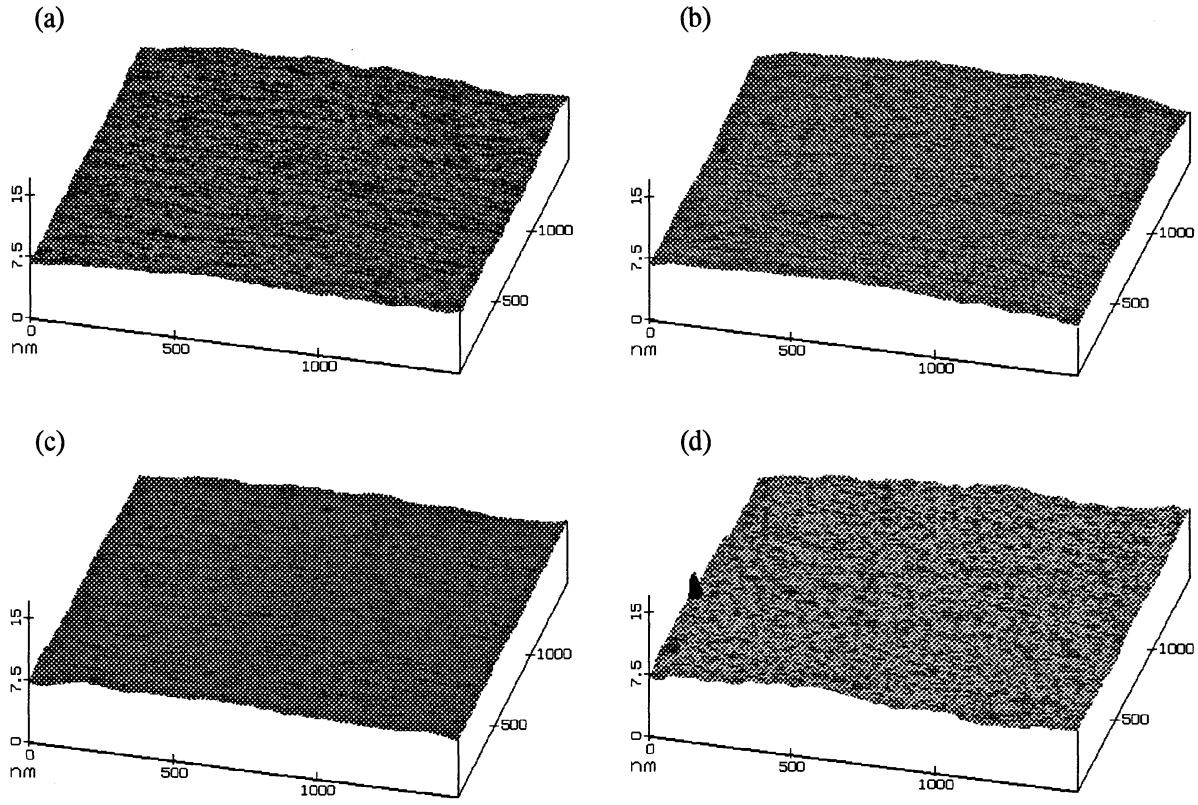

Fig. 1. AFM profiles of the bare fiber surface of fibers (a) A, (b) C, (c) D and (d) E, stripped in 40°C $FeCl_3$ for 10 minutes.

growth were in reasonable agreement. Fiber surfaces have been examined using AFM in an attempt to image the surface roughening caused by such a reaction. Fig. 1 shows AFM images of stripped surfaces of fibers A, C, D, and E for a 10 minute stripping time. Fig. 2 shows AFM images of fiber B after stripping for (a) 10 and (b) 20 minutes. Surface particles are apparent in Fig. 2(a) after stripping for 10 minutes but are mostly removed after 20 minutes. These particles are thought to be residual aluminum, and that the aluminum is more strongly bonded to fiber B than to the others, because of the greater difficulty in removing the last layers of the coating. However, provided there has been sufficient etching time, the fiber surfaces all appear smooth on the nanometer scale. Any slight differences in roughness that are apparent are artifacts of the AFM technique and are, anyway, not big enough to explain the large differences in strength. Application of the aluminum coating does not therefore cause widespread roughening of the fiber surface. The strength degradation is probably not caused by the interfacial reaction between aluminum and the glass. It is possible that the aluminum etches the glass at occasional sites which were missed by AFM, since the AFM scan area is small (~2.25 μm^2) compared with the effective specimen size in bending (~10^4 μm^2).[9] However, the longer stripping time needed for fiber B suggests that there may be a correlation between the higher strength of fiber B and the better coating adhesion.

Another possibility is that the grain structure of the metal coating may influence the fiber strength. Bogatyrjov *et al.*[14] reported that the microstructure of hermetic tin coatings controls the strength of tin-coated fibers. They found that the silica glass fiber with a tin coating with grains all in the same orientation, had a higher strength and a much narrower Weibull distribution than the same fiber coated with tin of random grain orientation. However, we found that the aluminum-coated fibers did not show a similar effect. Fig. 3 shows SEM images of the surface of the aluminum coating for the five fibers. The coatings of fiber C and E have similar "fish scale" microstructures, fiber C having the larger grain size (~3 μm, *c.f.* ~2 μm for fiber E). The

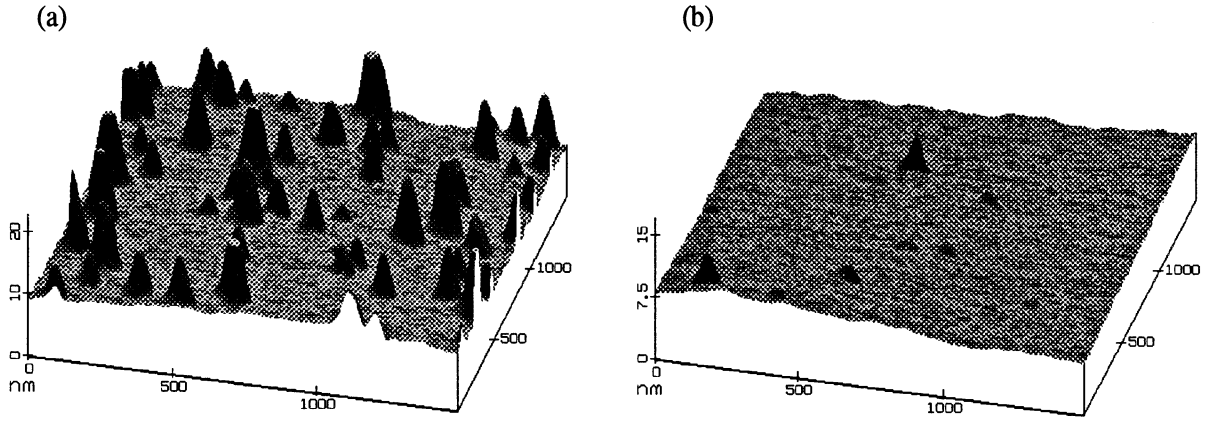

Fig. 2. AFM profiles of the bare surface of fiber B after stripped in 40°C $FeCl_3$ for (a) 10 minutes and (b) 20 minutes.

coating surface of fiber A was not smooth and had many small bumps. Fibers B and D had smooth coating surfaces except for some scratches. The grain structures of these three specimens have not yet been established. No particular relationship between the microstructure of aluminum coatings and fiber strengths was found.

The difference in sensitivity to grain structure observed for tin- and aluminum-coated fibers can be explained by Von Mises theory, which requires five independent slip systems in order to accommodate a general homogeneous strain by slip.[15] At room temperature, tin has a tetragonal structure with only four independent slip

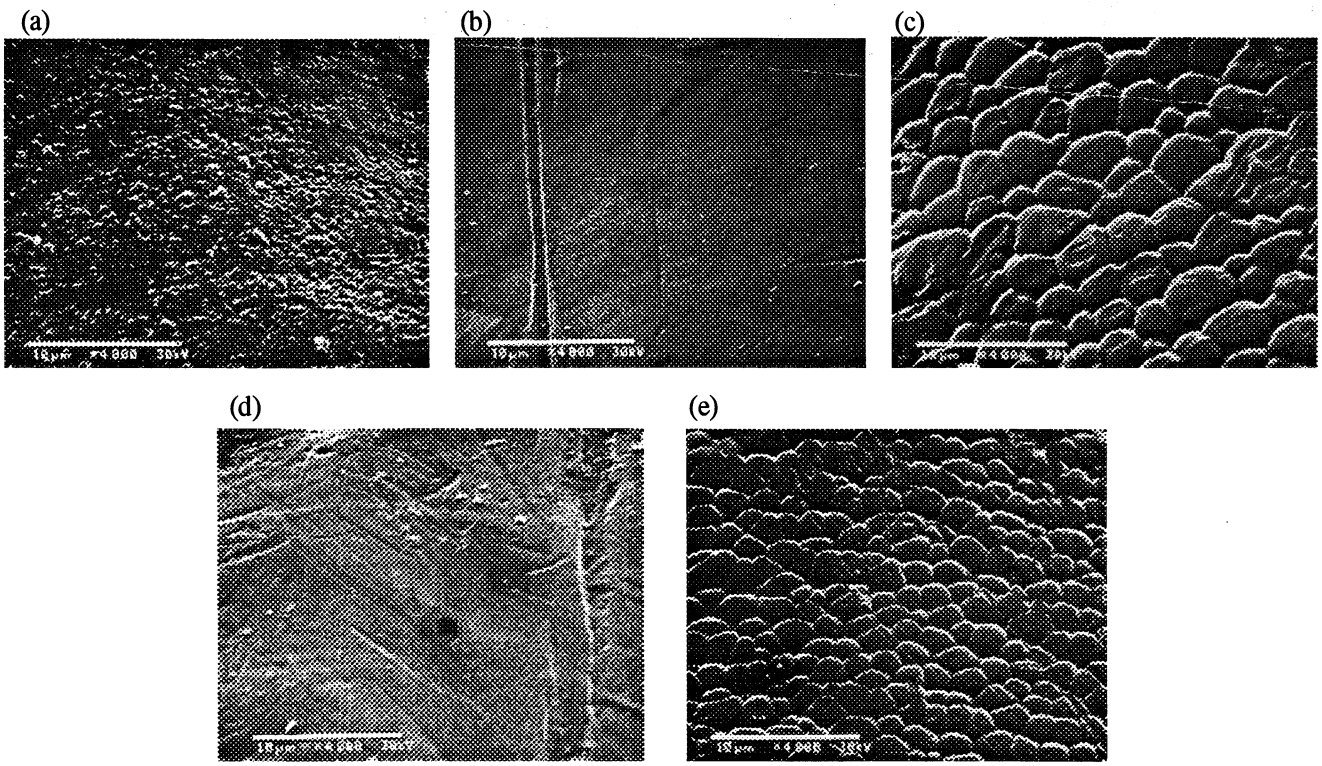

Fig. 3. SEM images of the aluminum surface of fibers (a) A, (b) B, (c) C, (d) D and (e) E. Scale bars are 10 μm long.

systems; it can only accommodate a general strain if all the grains are aligned.[14] However, aluminum has a face centered cubic structure with five independent slip systems and can accommodate strain for any grain orientation.

4. CONCLUSIONS

Five aluminum coated fused silica fibers from different sources had different strengths despite being manufactured using similar processes. The AFM results for stripped fibers did not show any roughening of the fiber surfaces nor any difference between the five specimens. SEM studies on the aluminum coatings showed that the microstructures were somewhat different; but no direct correlation between fiber strength and grain structure was identified. However, the coating on the strongest fiber, specimen B, was found to be the hardest to remove by chemical stripping. It is concluded that the coating adhesion may play a role in the strength of aluminum-coated fibers. The operating temperature and atmosphere during coating are expected to influence this adhesion.

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