Polyimide fibers for harsh environments



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Optical fibers in harsh environments

This document discusses the use of polyimide coating for high temperature environments. While silica glass can intrinsically withstand temperatures well beyond 800°C, the coating that surrounds the silica fiber degrades at a much lower operating temperature, making the coating the element that determines the maximum operating temperature of the fiber.

Harsh environments usually refer to extreme temperature and/or radiation level, and encompasses environments such as nuclear facilities, high energy physics laboratories, and space.









Leveraging 50+ years of continuous improvements made on standard telecom fibers, specialty optical fibers are now widely used in harsh environments for data transmission, sensing or in-vitro diagnosis.

Passive or active specialty fibers offer virtually unlimited design possibilities and can be optimized for UV, visible or NIR wavelengths through the careful selection of raw material, dopants and doping profile. In addition, most of the key characteristics that led to the wide development of optical fibers for telecommunications apply equally well to specialty fibers:

- Immunity to EM
- Lightweight and low volume
- Low loss
- High temperature resistance
- High bandwidth





Coating: a multi-purpose element

The development of high-quality coatings played a major role in making optical fibers practical for use in real life environments. The coating is a multi-purpose element, some of its key functions are listed below:

1. Protect the mechanical integrity of the fiber

Optical fibers without coating are extremely fragile, and direct contact of the glass with hard external surface can create microscopic flaws which will cause a fiber break when a critical size is reached. The coating acts as a protective mechanical layer which both reduces the creation of such defects and limits the growth of existing unavoidable micro-flaws.

2. Shield the silica glass from chemicals

It has been well documented in the literature that water vapor weakens the SiO_2 structure, enhancing crack propagation and potentially ultimately leading to a fiber break. The coating acts as a chemical barrier to prevent chemical compounds from reaching to the silica glass.

3. Limit micro-bending

Micro-bending comes from irregularities and stress applied on the fiber on the microscopic scale (hundreds of micrometers or less) and results in an excess loss. Micro-bending can be minimized with a combination of soft primary and hard secondary coatings that reduces the stress applied on the silica fiber.

Main types of coating

Most of the coatings in use nowadays are made of polymer. Polymer coatings are relatively easy to manipulate, provide good mechanical and chemical resistance and are very well suited to handle moderate temperatures found in outdoor applications.

Acrylate coating is the standard for telecom fibers, with a maximum operating temperature of +85°C. It remains to this day the preferred coating for standard environmental condition, but its relatively low maximum operating temperature and low radiation resistance are not suitable for harsh environments with high temperature and/or radiation levels.

High temperature acrylate, often referred as acrylate HT, was specifically developed to offer an extended operating temperature up to +150 $^{\circ}$ C with good radiation resistance. Finally, polyimide is the preferred choice for the most demanding applications, with a maximum operating temperature of +350 $^{\circ}$ C and excellent radiation resistance. It is worth noting that polyimide fibers typically have an outer diameter of only 150 - 160 μ m, compared to 250 μ m for acrylate coated fibers.

The table below summarizes the maximum operating temperature and radiation resistance of the main polymer coatings:



	iXblue code	Max temperature	Radiation resistance
Acrylate		+85 °C	Poor
High Temperature	-HT	+150 °C	Good
Acrylate			
Polyimide	-PI	+350 °C	Excellent

Additional information regarding the combined effect of temperature and radiation can be found in the publications referenced in footnotes 1 and 2. Metal coatings such as aluminum can handle long term temperatures beyond 400 °C and are currently under development to complete iXblue's fiber portfolio for high temperature applications.

Coating deposition

The coating is deposited during the drawing process before the bare silica fiber touches any hard external surface. This ensures a proper protection of the fiber before it touches the metallic drawing pulleys that pull the fiber at the bottom of the drawing tower.

For polymer coatings, the fiber goes through a bath of liquid polymer and exits through a die of calibrated diameter which sets the outer diameter of the coated fiber. The fiber is then cured in-line using a UV or thermal furnace to polymerize the coating. Depending on the coating material and recipe, several layers of coatings can be applied on top of each other to achieve the desired opto-mechanical characteristics. Each layer may have a different chemical composition and thickness.

² <u>Radiation resistant single-mode fiber with different coatings for sensing in high dose</u> environments



¹ Combined effect of radiation and temperature: towards optical fibers suited to distributed sensing in extreme radiation environments

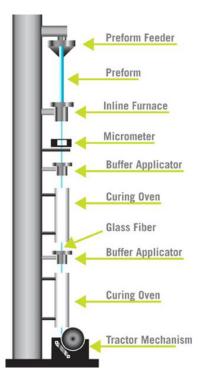
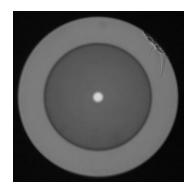


Figure 1: schematic representation of the fiber drawing tower, with 2 buffer applicators and curing ovens placed one after another to apply 2 layers of coating.

The outside coating diameter is measured in-line during fiber drawing, together with many other drawing parameters. These parameters are monitored during the draw for process control and saved in a database for QA and traceability.

The coating deposited should be free of any bubble or inclusion and should have a constant diameter along the fiber length. Achieving a high-quality coating relies equally on selecting the most adequate raw materials and on the deposition process (number of layers of coating, thickness of each layer, drawing speed, etc.).





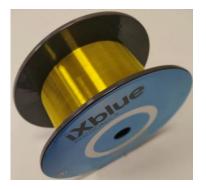


Figure 2: Typical fiber cross-section under microscope (top left), top view of a polyimide coated fiber under microscope (top right), and spool of polyimide fiber (bottom left).



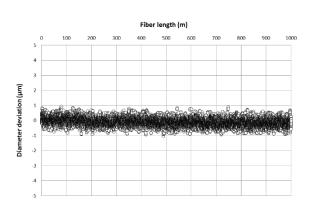
In addition to providing optimal mechanical and chemical resistance, a defect-free coating is also of paramount importance for Fiber Bragg Gratings (FBG) inscription using femtosecond laser. Given that the inscription is done through the coating, a smooth and high-quality coating is needed to achieve the desired FBG characteristics with a high yield. Dozens or hundreds of FBGs can be inscribed along the fiber length for distributed strain or temperature sensing. Specialty optical fibers designed for harsh environments are well suited for Structural Health Monitoring (SHM) or Distributed Temperature Sensing (DTS) of structures operating at extreme temperatures or under high radiation doses.

iXblue's Polyimide

iXblue is continuously expanding its portfolio of specialty optical fibers to offer solutions for harsh environments such as nuclear facilities, defense applications, high energy physics laboratories or space. Polyimide coating is now readily available for volume production following R&D, qualification, and industrialization phases. Some key characteristics of iXblue's polyimide fibers are summarized below:

Some key characteristics of polyimide coated fibers are shown below and in Figures 3, 4, 5 and 6.

- Coating diameter stability
 - o ±1 µm along the fiber
- Mechanical strength
 - \circ Compliant with GR-20-CORE Telcordia, dynamic fatigue coefficient n_d = 25
- Temperature resistance
 - 15 years at 250 °C
 - 1 year at 300 °C
 - o 1 month at 350 °C
- Outgassing for space or high vacuum applications
 - o Total Mass Loss (TML) < 1% (125° C during 24 hours at P < 10^{-4} Pa). Polyimide fibers have a TML two to threefold better than acrylate coated fibers and are therefore well suited for vacuum applications.



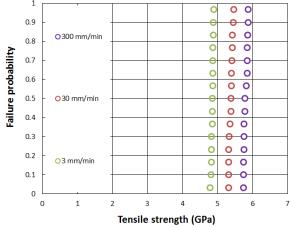
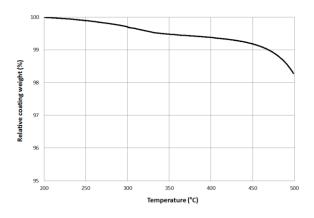


Figure 3: Variation of coating diameter along the fiber length. Each point corresponds to the average coating diameter over 15 cm.

Figure 4: Fiber gauge of 0.50 m.
Obtained mechanical properties comply with
GR-20-CORE Telcordia norm.





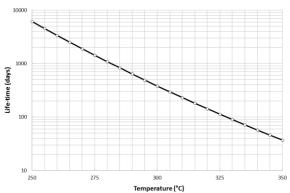


Figure 5: Mass loss of the polyimide coating as a function of temperature.

Figure 6: Lifetime prediction for isothermal degradation under air, based on coating weight loss of 20%.

For cryogenic temperatures, the fiber should remain still once installed in the cryogenic environment because of the brittle nature of polymers well below their glass transition temperature. Nevertheless, tests performed on polyimide coated fibers cooled at liquid nitrogen temperature (-196 $^{\circ}$ C) for 24 hours showed no degradation of the optical transmission and excellent resistance to mechanical traction.

Polyimide is a relatively hard coating with excellent temperature resistance. Conversely, the high Young modulus of polyimide transfers more stress on the fiber and microbending is more pronounced than with a softer coating such as acrylate. The optical loss of polyimide fibers is therefore sensitive to the environmental conditions in which the fiber is being used, whether the fiber is spooled, in a cable, or simply coiled manually.

Striping polyimide

Similarly, the hard nature of Polyimide makes it more difficult to strip than acrylate coatings. Regular mechanical and thermal stripping tools are designed for acrylate fibers and should be avoided for polyimide fibers.

The polyimide coating can be removed with alternative thermal methods or using chemicals. For instance, it is possible to use a CO_2 laser, a flame from an oxygen/hydrogen torch, or an electric arc to expose the polyimide to temperature beyond which it degrades. These methods preserve the mechanical resistance of the silica fiber but are however relatively difficult to implement. For this reason, it remains relatively common to strip polyimide using the flame of a lighter, though this method is less reproductible and more prone to affect the mechanical characteristics of the silica fiber.

To strip longer lengths, the polyimide fiber can be placed in an oven at a temperature beyond 600°C for 1 hour, or at a higher temperature to speed up the removal of the polyimide. Alternatively, a bath of hot sulfuric acid (100°C) will efficiently remove the polyimide coating in a couple of minutes, though this method requires appropriate precautions related to the use of hazardous chemicals.



Conclusion

Polyimide provides improved thermal resistance compared to acrylate and high temperature acrylate coatings. With a maximum temperature of +350 $^{\circ}$ C, polyimide is the preferred choice for high temperatures applications. For operating temperatures below +150 $^{\circ}$ C, high temperature acrylate is a more cost-effective solution.

Fibers used in high temperature environment may sometimes also be exposed to radiations, for example in nuclear facilities. The table below summarizes the recommended fiber depending on the temperature and radiation levels.

Øclad = 125 µm, NA = 0.14	Temperature ≤ 150 °C	150 °C ≤ Temperature ≤ 350 °C
Low-moderate radiation levels	IXF-SM-1550-125-0.14- <u>HT</u>	IXF-SM-1550-125-0.14- <u>PI</u>
High radiation levels	IXF- <u>RAD</u> -SM-1550-0.14- <u>HT</u>	IXF- <u>RAD</u> -SM-1550-0.14- <u>PI</u>

Polyimide coating can be applied indifferently on single-mode, multimode or custom fibers, do not hesitate to contact your iXblue sales representative if a reference is not available from our catalog.

With more than 20 years of experience in manufacturing specialty optical fibers, iXblue has all the expertise and tools to address custom developments and high-volume requirements.

