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**Silica-silica Polyimide Buffered Optical Fibre Irradiation and Strength Experiment
at Cryogenic Temperatures for 355 nm Pulsed Lasers**

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Abstract

A controlled UV-light delivery system is envisioned to be built in order to study the stability properties of superconducting strands. The application requires a wave guide from room temperature to cryogenic temperatures. Hydrogen loaded and unloaded polyimide buffered silica-silica 100 μm core fibres were tested at cryogenic temperatures. A thermal stress test was done at 1.9 K and at 4.2 K which shows that the minimal mechanical bending radius for the fibre can be 10 mm for testing (transmission was not measured). The cryogenic transmission loss was measured for one fibre to assess the magnitude of the transmission decrease due to microbending that takes place during cooldown. UV-irradiation degradation measurements were done for bent fibres at 4.2 K with a deuterium lamp and 355 nm pulsed lasers. The irradiation tests show that the fibres have transmission degradation only for wavelengths smaller than 330 nm due to the two photon absorption. The test demonstrates that the fibres are suitable for the cryogenic UV applications with 355 nm and 70 μJ pulsed lasers.

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ABSTRACT

A controlled UV-light delivery system is envisioned to be built in order to study the stability properties of superconducting strands. The application requires a wave guide from room temperature to cryogenic temperatures. Hydrogen loaded and unloaded polyimide buffered silica–silica 100 μm core fibres were tested at cryogenic temperatures. A thermal stress test was done at 1.9 K and at 4.2 K which shows that the minimal mechanical bending radius for the fibre can be 10 mm for testing (transmission was not measured). The cryogenic transmission loss was measured for one fibre to assess the magnitude of the transmission decrease due to microbending that takes place during cooldown. UV-irradiation degradation measurements were done for bent fibres at 4.2 K with a deuterium lamp and 355 nm pulsed lasers. The irradiation tests show that the fibres have transmission degradation only for wavelengths smaller than 330 nm due to the two photon absorption. The test demonstrates that the fibres are suitable for the cryogenic UV applications with 355 nm and 70 μJ pulsed lasers.

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1. Introduction

Fast and well defined energy deposition to study the stability properties of superconducting strands is envisioned to be used in the future. Based on earlier successful experiments that can be found in the literature [1] it was decided to use laser energy deposition to study the phenomena. The earlier experiments were done to study the thermal stability of NbTi-strands, hence the pulse width was in the order of tens and even hundreds of milliseconds [2]. The laser wavelength was in IR-range which is very poorly absorbed by copper at 4.2 K [3].

In future studies the main objective is in the magneto-thermal instabilities of Nb₃Sn-strands [4]. The intention is to be able to define the minimal perturbation energy to trigger the magneto-thermal instability in superconducting strands (minimum trigger energy, MTE) as accurately as possible. This means that the pulse width must be smaller than 1 μs because the energy deposition needs to be faster than the thermal diffusion time. The characteristic thermal time constant in a 1 mm wide slab of copper is 1 μs

at 6 T [5]. The Q-switched lasers provide a very fast and powerful pulse [6]. Hence, a diode pumped passively Q-switched frequency tripled solid state laser 355 nm from CryLas™ was chosen to generate the required pulse. It is able to provide a 1 ns and 70 μJ pulse with a maximum repetition rate of 100 Hz. This wavelength is absorbed very well by copper at room temperature as well as at 4.2 K [7,8]. It is for example used to micromachine copper [9]. The laser can be coupled with an optical fibre whose diameter is bigger than 70 μm . The controlled light delivery of high-power pulsed UV-lasers at 355 nm wavelengths in order to illuminate strands under test at cryogenic temperatures requires that the fibre has to withstand a steep temperature gradient, from room-temperature to cryogenic temperatures.

The objective of this study was to find a suitable optical fibre for the laser. The chosen fibres should be adapted to cryogenic temperatures with a bending radius of 30 mm in order to gain flexibility in mechanical design of the upcoming experiments. The UV-enhanced hydrogen loaded and unloaded silica–silica optical fibres are tested. The hydrogen is used to heal irradiation induced damages in silica as shown in deep UV-region below 330 nm wavelengths, after irradiation with deuterium lamps [10], with pulsed excimer-laser or 4th harmonic Nd-YAG lasers [11] and with Gamma-sources [12]. It is expected to see microbending due to the cooldown and irradiation induced transmission degradation. The fibres might become more fragile in cryogenic temperatures which makes it possible to have mechanical breakage due to thermal stress or opto-acoustic shock.

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Table 1
Characteristics of the fibre [13].

Characteristics	
Product descriptor	FVP100110125
Core material	Silica, high OH (with and without hydrogen)
Core diameter	100 μm
Buffer material	Polyimide
Buffer diameter	125 μm
Cladding diameter	110 μm
Cladding material	Silica, F-doped
Numerical aperture	0.22 ± 0.02
For 10 m long fibre, transmission	From 220 nm up to 1200 nm

2. Material and methods

The application needs a UV-enhanced fibre, therefore hydrogen loaded and unloaded polyimide buffered silica–silica fibres (Table 1, [13]) were chosen to be tested. Three types of tests were done on the fibres: thermal stress tests, irradiation tests and a cryogenic transmission loss test.

The thermal stress test consists in immersing the optical fibre samples into liquid helium at 1.9 K to verify if it can adapt mechanically to the cold environment without developing breakages or cracks. In order to assess the presence of cracks, the fibre samples were examined, once warmed up at room temperature, using an optical microscope. Three unloaded (hydrogen) fibre samples were tested; each of them had a circular geometry with a different bending radius (10 mm, 20 mm and 30 mm). The cooling procedure for the thermal stress test is shown in Table 2. For future tests, these experiments with significantly lower bending radii are sufficient with 3 h duration at 1.9 K.

The irradiation tests were done to assess the degradation induced by UV-irradiation on hydrogen loaded and unloaded fibres at cryogenic temperatures. For this purpose, UV-light was injected into the fibre for several hours and every few minutes the transmission spectrum was measured using a deuterium lamp that provides a UV-spectrum which has an FOS-signal peak at 250 nm, which is generated by the spectral transmission of the spectrometer (white light interference has to be taken into account), the transmission of the fibre and the emission spectrum of the deuterium lamp with a maximum around 190 nm. For some samples the deuterium lamp was used not only to measure the transmission spectrum but also to damage the fibre. When the transmission spectrum is measured, the lamp is activated in as short time as possible to minimize the degradation in the fibre. While, when the deuterium lamp is used to measure and degrade, it is activated continuously for a long period of time. For degrading the fibre, two Q-switched UV lasers with 1 ns pulse width and 355 nm wavelength were also used. The first laser provides a 1 μJ pulse energy with a repetition rate of 25 kHz. The second laser provides a 70 μJ pulse energy with a repetition rate of 100 Hz. This laser was used to check the effect of the opto-acoustic shock which is dependent on the pulse energy. The characteristics of the light-sources are shown in Table 3. The fibres were fed into the cryostat and back to room temperature through a flange. Inside the cryostat, four loops were made with the fibre. Two different bending radii, 25 mm and 15 mm, were used. The circular parts of the fibres were immersed in liquid helium bath and the fibres were irradiated while being at 4.2 K. They were in direct contact with liquid helium at 1 atm pressure. The fibre diameter is very small, only 125 μm . Thus, it was sufficient to drill 150 μm holes in the flange and the fibres were fed through them. A sketch of the experiment is shown in Fig. 1. Two types of samples were tested. One loaded and one unloaded fibres with loops of 15 mm radius were tested

Table 2
Thermal strain test procedure.

Procedure	Duration
Cooldown from 270 K to 77 K	1 h
Cooldown from 77 K to 4.3 K	1.3 h
Staying at 4.3 K	3.5 h
Cooldown from 4.3 K to 1.9 K	0.5 h
Staying at 1.9 K	3 h

Table 3
Characteristics of the light sources used in the measurements. E_p and t_p are the pulse energy and width, respectively.

Name	λ (nm)	t_p (ns)	E_p (μJ)	Rep. rate (Hz)
Laser 1	355	1	1	25 k
Laser 2	355	1	70	100
D ₂ -lamp	UV spectrum	–	–	Cont.

Fig. 1. Sketch of the set-up for the irradiation tests. A is the source, B is the focusing lens, C is the fibre, D is the cryostat (contains liquid helium at 4.2 K temperature and at 1 atm pressure), E is the helium level line and F is the detector. There were two fibre lengths, short/long, which are divided in three parts: 600 mm, 950 mm and $4 \times 15/4 \times 25$ mm. First mentioned part is outside of the cryostat, second is to/from the loop and the last is part with the four loops. Thus, the whole lengths are: 3500 mm and 3700 mm for the short and long, respectively.

with the first laser. Two fibres identical to the fibres before were irradiated with the deuterium lamp. One partly loaded fibre with loop of 25 mm radius was tested with the second laser. The samples regarding the irradiation tests are shown in Table 4. Hydrogen diffuses out from the loaded fibres if they are kept a long period of time at room temperature [14,15]. All the fibres were installed at the same time and the tests last for one week. Sample L2 was measured last, the part outside of the cryostat of the sample L2 was losing hydrogen during the tests. Therefore, L2 is considered only partly loaded. The L2 was measured mainly to show that the fibres did not break due to the opto-acoustic shock of the more energetic 70 μJ pulse. The result of this test should be applicable to loaded and unloaded fibres because the hydrogen has no effect on the opto-acoustic shock. The samples were tested according to Table 5.

The cryogenic transmission loss test was done to assess the ratio between the energy transmitted through the fibre when it is partially immersed in liquid helium at 4.2 K and the energy transmitted by the fibre at room temperature. This test was carried out by using the same setup as in irradiation tests with only one of the loaded samples (S1, Table 4); the experiment goal was to estimate the order of magnitude of the fibre degradation with respect to its transmission capabilities. The UV-spectrum was measured before

Table 4

Irradiation test samples. L2 is marked as partly loaded (x).

Sample	Loaded	Radius (mm)	Length (cm)	Light source
U1		15	350	D ₂ -lamp
U2		15	350	Laser 1
S1	x	15	350	D ₂ -lamp
S2	x	15	350	Laser 1
L2	x	25	370	Laser 2

Table 5

Irradiation test parameters.

Sample	Irrad. time (h)	P. count (Millions)	E_p (μ J)	t_p (ns)
U1	12	–	–	–
U2	12	1080	1	1
S1	6.4	–	–	–
S2	6	540	1	1
L2	9	3.24	70	1

and after the cooldown by using the deuterium lamp. During the cooldown the fibre was not irradiated.

3. Theory

There are five effects during the tests in the fibres: macro bending, thermal stress/microbending, opto-acoustic shock, UV-irradiation induced degradation and healing effect by hydrogen.

The transmission decreases in fibre when it is suffering of macrobending or microbending. These effects are due to the change in the reflection angle between the beam and the optical fibre [16]. The macrobending is caused by sharp bends in fibres where it is also capable of causing mechanical failures. The microbending is caused by the thermo-mechanical stress, which takes place at low temperatures [16]. The thermo-mechanical stress is due to the thermal contraction difference in the buffer, core and cladding layers.

The opto-acoustic shock is produced by the interaction of light with the fibre material. The magnitude of the effect is dependent on the pulse energy. The fibres might get more fragile during the cooldown and therefore a mechanical failure due to the opto-acoustic shock is possible.

There is attenuation in optical fibres which is due to the Rayleigh scattering and absorption of the photons. Typical UV attenuation of the 100 μ m silica-silica high OH content fibre is shown in Fig. 2. The attenuation in the fibres gets more significant at shorter wavelengths. The absorbed UV-light can break the Si–O–Si bonds into non-bridging oxygen hole centers (NBOHC, Si – O $\cdot$) and E'-centers (Si $\cdot$). They have absorption bands at 265 nm (4.7 eV) and 214 nm (5.8 eV) [17]. Moreover, with high intensities >10 MW/cm² and fibre lengths $\geq$ 3.5 m the attenuation is expected to be higher at wavelengths lower than 330 nm, which is due to two photon absorption [18]. At wavelengths lower than 200 nm attenuation is further induced by an unstructured OH-absorption tail [17]. In addition to nonlinear transmission due to two photon absorption, defects below 330 nm can be generated as well.

In silica, there are three different types of E'-centers called E' $_{\alpha}$, E' $_{\beta}$ and E' $_{\gamma}$ [19]. Their population is temperature dependent. This is because the recombination of the broken bonds depends on the movement of the mobile hydrogen atoms, which depends on the temperature [19,20]. It is possible to load the fibre with hydrogen in order to induce the healing process. The mobile hydrogen molecules are able to move in the glass and they transform the broken molecules to Si – OH + H – Si [17]. Therefore, it is expected that the hydrogen loaded fibres will have less degradation at lower wavelengths than the unloaded fibres. However, the recombina-

Fig. 2. Typical attenuation of the 100 μ m silica-silica high OH fibre. The result is from measuring and comparing the transmission spectra of a long and a short fibres by using the deuterium lamp.

Fig. 3. The deuterium lamp spectrum through the S1 loaded fibre before the cooldown and after the cooldown from room temperature to liquid helium bath temperature at 4.2 K.

tion process could slow down due to the cryogenic temperature and therefore the defect population would be higher than at room temperature. This would result in increased attenuation at 265 nm and at 214 nm wavelengths.

4. Results

In the thermal stress experiment none of the fibres broke and when examined with an optical microscope, no differences between unused and thermally cycled fibres were seen.

In Fig. 3 the results are shown for the cryogenic transmission loss of the S1 loaded fibre at 4.2 K and before and after the cooldown. The results show that the cooldown reduces the intensity throughout the measurement range. The cryogenic loss is approximately 25% and 35% for 355 nm and 250 nm wavelengths, respectively. The deuterium lamp irradiation test with the S1 loaded fibre is shown in Fig. 4, there was no UV irradiation induced degradation.

There was no degradation during the laser 1 irradiation test with the S2 loaded fibre. The deuterium lamp spectra before and after the irradiation were similar to the results in Fig. 4.

In Fig. 5 the results are shown for the deuterium lamp irradiation test with the U1 unloaded fibre at 4.2 K. The figure shows degradation in deep UV-light range but there is no degradation for wavelengths above 330 nm. After 6 h of irradiation there is approximately 28% degradation at 250 nm wavelength.

In Fig. 6 the results are shown for the laser 1 irradiation test with the U2 unloaded fibre at 4.2 K. The figure shows that there is some degradation in the deep UV-light range after the laser irradiation but no degradation above 330 nm wavelength.

In the laser 2 irradiation test with the L2 partly loaded fibre. The deuterium lamp spectrum with the L2 was not measured due to

Fig. 4. The figure shows deuterium lamp spectrum through the fibre before the irradiation and after the irradiation at 4.2 K.

Fig. 6. Deuterium lamp spectrum through the sample U2 before and after 12 h of irradiation with the laser 1.

Fig. 5. U1 unloaded fibre during deuterium lamp irradiation at 4.2 K.

Fig. 7. Summary for all the irradiation losses for 250 nm and 355 nm wavelengths. The figure represents the transmission after irradiation in respect to the initial transmission at 4.2 K.

some technical problems. However, it was measured with a photodiode that the intensity at 355 nm during the laser irradiation did not change.

5. Discussion

The thermal stress test showed that the fibre is mechanically able to adapt to cryogenic temperatures. In our tests, no breakages were observed with the 100 μm core fibres, even with 10 mm bending radii over 3 h at 1.9 K; these bending radii are significantly smaller compared to the necessary bending radii needed in our future testing equipment for superconducting strands.

The cryogenic transmission loss test showed that there is degradation in the sample S1 which is observed throughout the measured wavelength range. This is due to microbending that happens in the fibre. Materials contract differently when the fibre is cooled down: the silica-based core and cladding in contrast to the Polyimide coating have significantly different thermal expansion coefficients. However, the optical loss is 1.8 dB at 250 nm and 1.2 dB at 355 nm wavelength.

The irradiation test with deuterium lamp and with the laser 1 shows that in the unloaded samples U1 and U2, respectively, the transmission degrades in time at wavelengths below 330 nm as expected due to the two photon absorption generating E'-centers (at 215 nm) and NBOH-centers (at 265 nm). However, in loaded samples S1 and S2 there was no optical degradation. The difference between the loaded and the unloaded samples is due to the healing effect of hydrogen molecules, as described above.

The results of L2 shows that the 70 μJ pulses are not energetic enough to degrade the fibre due to the opto-acoustic shock at 4.2 K. No fibre breakage was observed over 9 h even in the bent

fibre section with a bending radius of 25 mm. Due to out-gassing of hydrogen, the spectral transmission changes in the non-cooled fibre section outside the cryostat [14]. The result is applicable to unloaded fibres since hydrogen loading should not play any role on opto-acoustic shock.

The summary of all the irradiation losses is shown in Fig. 7. The figure shows that the 355 nm transmission does not suffer from irradiation degradation unlike the 250 nm transmission. The 250 nm values cannot be considered as final values because the irradiation could generate a higher optical and mechanical damage in the fibres with increasing testing time. Most critical, mechanical damage can be occurred in the bent section of the fibre. Therefore, the maximum testing period and the minimum bending radius in the final set-up for strand tests have to be committed.

6. Conclusions

From the mechanical point of view, the polyimide buffered silica-silica fibres can be bent with radii 10 mm at 1.9 K. This fact was confirmed with the thermal stress experiment.

Due to the microbending, the cryogenic losses in bent silica-silica fibres can be in the order of tens of percents with fibre lengths in the order of meters. The losses are dependent on the wavelength as shown for two wavelengths.

It was known that at room temperature the fibres suffer from UV-induced degradation due to UV-irradiation; using hydrogen, the loaded fibres will be healed in parallel during the UV-irradiation. For the first time, it was confirmed to be true in hydrogen loaded fibre at 4.2 K. The results show that after 1080 million pulses the laser 1 only degrades the transmission of the non-loaded fibre for wavelengths lower than 330 nm due to two photon

absorption leading to 178 nm light. If the fibres are hydrogen loaded, no spectral degradation has been obvious, at all. It was demonstrated that both pulsed lasers can be used with 100 μm core silica–silica optical fibres at 4.2 K without optical degradation; in addition the 70 μJ pulse is not strong enough to mechanically break the fibre due to the opto-acoustic shock.

So far, the minimum bending radius at 4.2 K was 25 mm when the fibre was irradiated with the laser 2. With the laser 1 no breakage or mechanical damage has been observed in the test fibre over an irradiation period of 12 h with a bending radius of 15 mm in the cryostat.

Based on these fibre studies the unloaded fibres can be efficiently used in the laser light delivery system that is envisioned to be used in the proposed measurement system for studying the stability properties of superconducting strands. The fibres will be coupled with a laser similar to the laser 2 that was used in the above discussed experiments.

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References

- [1] Thrillaud F. Etude de la stabilité thermoelectronique des conducteurs supraconducteurs a basse temperature critique et contribution a l'etude de la stabilité thermoelectrique des supraconducteurs a haute temperature critique. Ph.D. thesis. Institut nationale polytechnique de Grenoble; 2005.
- [2] Thrillaud F, Ayela F, Devred A, Fratini M, Leboeuf D, Tixador P. A novel technique for minimum quench energy measurements in superconductors using a single-mode diode laser. *Cryogenics* 45 (9).
- [3] Biondi MA, Guobadia AI. Infrared absorption of aluminum, copper, lead, and nickel at 4.2 K. *Phys Rev* 166 (3).
- [4] Bordini B, Rossi L. Self field instability in high-jc Nb3Sn strands with high copper residual resistivity ratio. *IEEE Trans Appl Supercond* 19 (3).
- [5] Wilson MN. Superconducting magnets. Oxford university press; 1983.
- [6] Milonni PW, Eberly JH. Laser physics. Wiley; 2010 (Ch. 6).
- [7] Biondi MA. Optical absorption of copper and silver at 4.2 K. *Phys Rev* 102 (4).
- [8] Koch EE, Krafka C, Lynch DW, Weaver JH. Optical properties of metals. Eggenstein-Leopoldshafen: Karlsruhe Fachinformationszent. Kernforsch. Zentralstelle At. Energ.; 1981.
- [9] Zhang W, Yao YL, Chen K. Modelling and analysis of UV laser micromachining of copper. *J Adv Manuf Technol*.
- [10] Klein K-F, Schließmann P, Hillrichs G, Belz M, Boyle WJO, Grattan KTV. UV-stabilized silica based fiber for application around 200 nm wavelength. *Sens Actuat* 1997;B38–39:305–9.
- [11] Karlitschek P, Hillrichs G, Klein K-F. Influence of hydrogen on the colour center formation in optical fibers induced by pulsed UV-laser radiation; part 1: all-silica fibers with high-OH undoped core. *Optics Commun* 1998;155:376–85.
- [12] Assmus J, Klein K, Nelson G, Clarkin J, Pross H, Kiefer J. Improvements of gamma-induced transmission damage in the deep UV-region. In: International conference on ageing studies and lifetime extension of materials paper 6.2.
- [13] P. Technologies, Data sheet for silica/silica optical FV fibres. World Wide Web electronic datasheet; 1998 <<http://www.datasheet.org.uk/FVP100110125-datasheet.html>>.
- [14] Klein K-F, Kaminski R, Hüttel S, Kirchhof J, Grimm S, Nelson G. Lifetime improvements of UV-improved fibers for new applications. In: SPIE-Proceedings BiOS'98 3262C; 1998. p. 150–60.
- [15] Klein K-F, Arndt R, Hillrichs G, Ruetting M, Veidemanis M, Dreiskemper R. UV-fibers for applications below 200 nm. In: SPIE-Proceedings of BiOS'01, vol. 4253; 2001. p. 42–9.
- [16] Crisp J, Elliott B. Introduction to fiber optics. Newnes; 2005 (Ch. 6).
- [17] Khalilov VKh, Klein K-F, Belmahdi J, Timmerman R, Nelson G. High-OH fibers with higher stability in the UV-region. In: SPIE-Proceedings 6083 (8).
- [18] Taylor RS, Leopold KE, Brimacombe RK, Mihailov S. Dependence of the damage and transmission properties of fused silica fibers on the excimer laser wavelength. *Appl Optics* 27 (15).
- [19] Griscom DL. Defect structure of glasses. *J Non-Cryst Solids* 73:51–77.
- [20] Griscom DL. Thermal bleaching of X-ray-induced defect centers in high purity fused silica by diffusion of radiolytic molecular hydrogen. *J Non-Cryst Solids* 68:301–25.