EXTERNAL REPORT
SCK•CEN-ER-168

EU/RF collaborative tasks on ITER diagnostics – EU Contribution to Optical Fibre Development

TW5-TPDS-DIARFB-Deliverable EU-9.1

EFDA Contract 05-1324

Benoit Brichard

October, 2010

SCK•CEN
Boeretang 200
BE-2400 Mol
Belgium

BSU/VMa/AWo/2011-010

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Background

The main objective of the task was to setup a dedicated facility to fabricate radiation resistant large core optical fibres loaded with very high hydrogen content. The fabrication of the hydrogen-loading facility was contracted to TRINITI Moscow from the Russian Federation. The main specifications and objectives of the task was defined at the Kick-Off meeting in Moscow, February 2006:

The present EU/RF collaborative task focuses on the supply of large-core (600 μm), metal-jacketed, hydrogen-loaded optical fibres made of KU1 glass

The role of SCK•CEN was defined at page 38 of the Annex A of the EFDA contract/05-1324 which mainly consisted in acting as advice role in the preparation of future irradiation tests (not part of the present contract) to be performed on the delivery of the radiation-hard fibres (under the responsibility of the Russian Federation). The SCK•CEN technical responsible in charge of the task attended the Kick- Off Meeting at the Kurchatov Institute, Moscow to establish in concertation with the RF team the specifications of the task. The SCK•CEN technical responsible also attended the first progress meeting organized remotely by EFDA. The intermediate report [1] (reproduced in annex) from SCK•CEN reported on the very first irradiation test on large KU1 fibres irradiated in BR2 and carried out outside and before the scope of the present task. Additional information was also later reported in the SCK•CEN Fusion Annual report of 2008 with the long term irradiation resistance of 200 µm aluminium-coated hydrogen-loaded optical fibres.
Unfortunately, a considerable delay arose from the Russian Federation side preventing to deliver radiation-hard metal-coated large core fibres as originally foreseen.

The present report aims to document (with appropriate references) the existing knowledge (scientific and technical) gained at SCK•CEN on the radiation resistance of hydrogenated fibres in order to close officially the present EFDA contract.

Purposes of radiation hard fibres

Saturation of silica glass with hydrogen reduces, in a very efficient way, the build-up of radiation-induced absorption (RIA) in the visible part of the spectrum (VIS), ie where the fibres are the most radiation sensitive. Hydrogen-loading approach was mainly driven by the need for very rad-hard optical fibres to be integrated in plasma spectroscopy diagnostic systems like MSE or CXRS. The previous work demonstrated (see below for a brief review) the feasibility for medium size core of 200 µm.
Nevertheless, there is still a need to overcome the technological difficulty to produce large core hydrogen-loaded optical fibres. Production of high hydrogen-content medium core optical fibres (200 µm) was prepared before 2005 by FORC/TRINITI at "low" pressure and temperature conditions requiring several weeks of hydrogen soaking in a dedicated autoclave to reach at least 10^{19} H_2/cm^3. In the same loading conditions, a fibre three times larger would require ten times more diffusion time putting the fibre fabrication process in the range of several months depending on the desired H_2 concentration in fibres. This effect can be appreciated at Fig 1 showing the hydrogen content versus diffusion time in bare 200 and 600 µm fibres (hence without aluminium coating !) soaked at different temperatures and pressures.

To reduce the fabrication time to acceptable time, it was decided to build an autoclave (TRINITI, Troisk, Moscow) able to load at 300°C, 300 bars and to produce large core hydrogen-loaded optical fibres for later irradiation testing. Up to now, those fibres have not been produced yet.

In the following, we summarize the outcome of the previous works carried out on the 200 µm fibre prototypes delivered under the auspices of the EFDA Irradiation Program on Ceramics (IRRCER) [2,3]. We also report on the ageing effect on those fibres irradiated again 2 years later.

Previous works and studies of medium core optical fibre (200 µm)

Hydrogen is a light element well-known to radically modify the optical properties (absorption and refractive index) in non-irradiated silica optical fibres. Hydrogen improves the optical transmission in visible (VIS) spectrum by reducing the as-drawn induced defects while, at the same time, it can considerably degrades the optical transmission in the infrared (IR) spectrum (>1000 nm). In addition, hydrogen is also able to reduce the formation of the RIA in the VIS when those hydrogenated fibres are exposed to radiation. The physical reason is now well understood [4]. Hydrogen blocks the formation of free radicals, which play an active role in the long-term RIA developing in VIS. In this spectral range, the main contributor to RIA in gamma-irradiated fibres is the Non-Bridging Oxygen Hole Centre (NBOHC). The blocking effect of hydrogen occurs through the cracking of the molecular hydrogen at the NBOHC sites (≡Si-O$^{\bullet}$), converting this later in OH groups which absorbs at 1.38 µm, ie outside the VIS window, according to the following mechanism

$$\begin{aligned} H_2 &\rightarrow 2H \\ \equiv Si-O^{\bullet} + H &\rightarrow \equiv Si-OH + 0.4\,\text{eV} \end{aligned}$$

This reaction is exothermic with a small activation energy of 0.1 eV.

The experimental demonstration of such mechanism has been reported in the literature (eg see Fig. 2 for irradiated KS4V 200 µm fibre). In the frame work of the EFDA Irradiation Program on Ceramics (IRRCER program), the Fiber-Optic Research Center in Moscow (FORC) provided SCK•CEN with prototypes of 200 µm diameter aluminium-coated fibres with high hydrogen content for radiation testing. The fibres were irradiated in various conditions, under gamma-rays, pulsed X-rays and fission reactor radiation. We briefly summarize the outcome of these irradiation tests performed on the 200 µm Al-coated hydrogen-loaded fibres provided by FORC, further details of the work can be found in the cited references.

- *Diffusion-limited energy-activated mechanisms*:
  The mechanism described above has been experimentally confirmed. Hydrogen acts on the NBOHC sites through diffusion-limited process and is only activated upon energy deposition in the fibres. Hydrogen diffusion ceases when irradiation stops. When hydrogen is exhausted RIA develops again quickly. At high dose (>MGy), we observed additional RIA degradation below 400 nm in hydrogen-loaded fibres compared to the unloaded counterpart underlying a different mechanism [5].

- *Reduction of transient RIA*:
  Beside the blocking effect of free radicals (as NBOHCs observed in the KU1 glass fibres) hydrogen can significantly reduce the transient RIA (of different nature) as observed in the glass fibres like KS4V (see Fig 2, [4]) and STU.

- *Pulsed X-rays and fission reactor irradiation*

The protection effect of hydrogen works even at extremely high dose-rate conditions, like X-ray pulses (>10^6 Gy/s) or at high total integrated dose such as seen in mixed gamma-neutron radiation field of fission reactors (>MGy ; >10^{17} n/cm^2). E.g. see Fig 3&4, also [5,6].

- *Performance of the hydrogen-loading conditions*

Using aluminium-coated fibres prepared from pure silica glass preforms and loaded in excess of 10^{19} n/cm^2 allows to fulfill very easily the specifications in term of radiation resistance of fibres for ITER diagnostics. Reactor tests showed a reduction of the RIA by one order of magnitude in the 450-650 nm wavelength range (VIS) [5].

- *Ageing effect of 200 µm*

One important question is to estimate how long a fibre can be protected by the hydrogen treatment. This question was never addressed however in details. In the present contract framework, we performed one additional gamma irradiation test two years after the loading using the remaining few meters of hydrogen-loaded STU 200 µm fibre produced by FORC. The fibre was stored on the shelf during this period at ambient temperature. The irradiation experiment was carried out in three steps at a slightly reduced dose-rate, 4.2 Gy/s (instead of 6 Gy/s as used in the first test) and counted three successive irradiations up to about 2.5 MGy.

Fig. 5 (a) and (b) display, as a function of the wavelengths and the dose, the RIA in the as-received hydrogen-free STU sample (marked 1) and its hydrogenated fibre version (marked 2) irradiated 2 years later. On the same graph (Fig. 5) we inserted the RIA curves of the as-received hydrogenated STU irradiated originally (marked 1). Fig. 5 hence allows to appreciate the storage ageing effect of hydrogen-loaded fibres.

We see that the RIA response of the older hydrogenated STU (marked (2)) at 630 nm is still below the hydrogen-free STU fibre sample but higher than the first sample. Clearly, the fibre has lost some hydrogen during the shelf period leading to a less efficient protection during irradiation. Nevertheless the radiation response still remains satisfactory, being quite close to the first irradiation, underlying the good performance of aluminium coating to retain most of the hydrogen. It would be also instructive from technological point of view to assess the ageing over several years to better assess its impact on the performance of the hydrogen-treated aluminium-coated fibres.

Conclusions

Hydrogen-loading is a powerful method to protect optical fibres against darkening in a large range of irradiation conditions [8]. Taking into account that all the irradiation tests are accelerated, i.e. involving high dose-rate conditions, and knowing that high dose-rates induce more RIA degradation in the fibres, we can infer that all our radiation tests are conservative and that hydrogen-loaded fibres can withstand the ITER radiation field to a large extent. The major difficulty is now in the technological fabrication of hydrogenated large-core fibre bundles. Such fabrication has not been demonstrated yet.

Endlessly ultimate radiation resistance in fibres could be thinkable by feeding the fibres constantly with a small amount of hydrogen. The approach could be envisaged using microstructured fibres containing air holes (porous medium) enhancing greatly and *in-situ* the hydrogen diffusion. Such an idea was demonstrated in a short BR2 irradiation test and already reported in the EFDA documentation.

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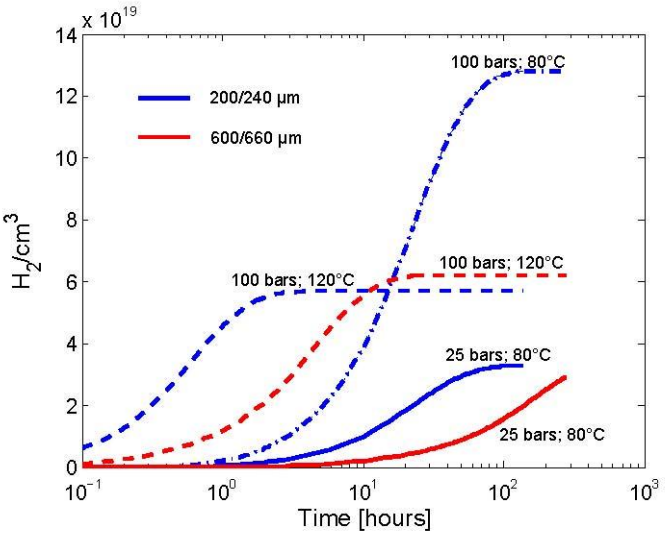


Fig 1: Theoretical hydrogen content in bare fibres as a function of pressure, temperature and time

RIA and Transient RIA in KS4V 200 µm fibre irradiated with gamma-rays

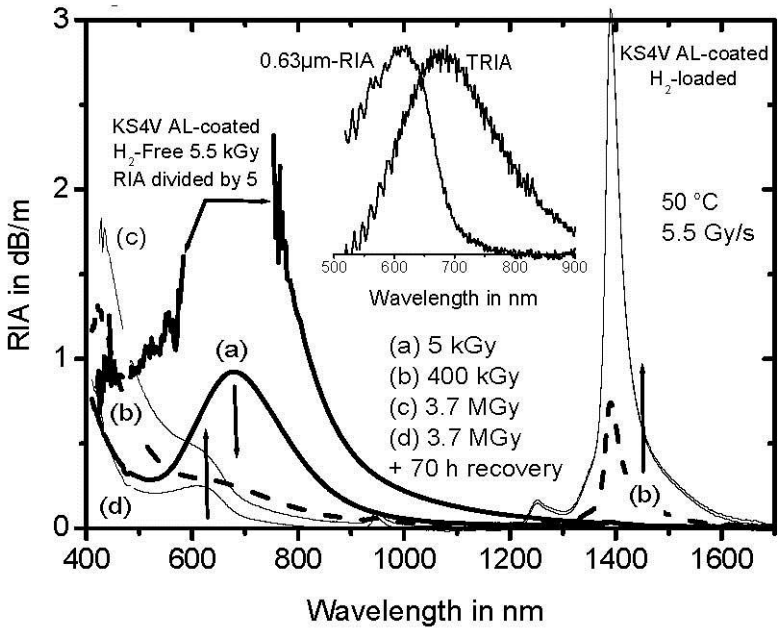


Fig 2: Decrease in the Transient RIA (TRIA) at 700 nm followed by the RIA increase at 630 nm as a function of the dose in H_2 treated aluminium-coated fibres. For comparison the corresponding TRIA in H_2-free aluminium coated KS4V fibre is also shown with a reduced factor of 5. The inset compares the shape of the TRIA and 0.63 µm-RIA in normalized axis when influence of the UV tail is removed.

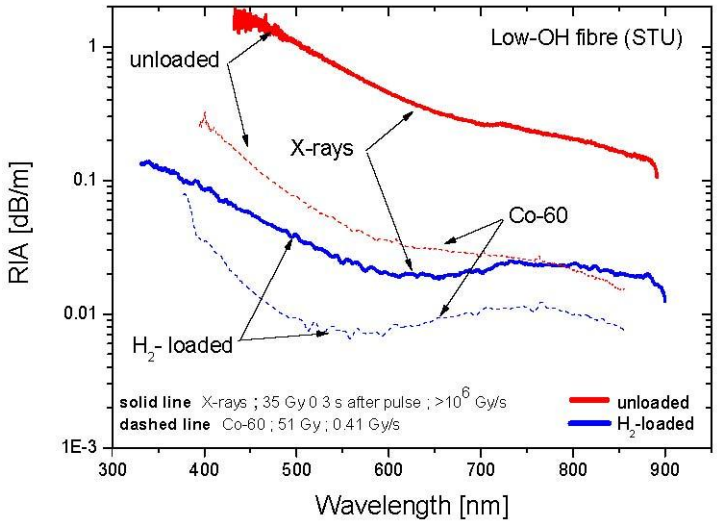


Fig 3: Comparison of the pulsed X-ray (solid lines) and steady state ^{60}Co (dashed-lines) irradiation in hydrogenated and untreated fibres [5].

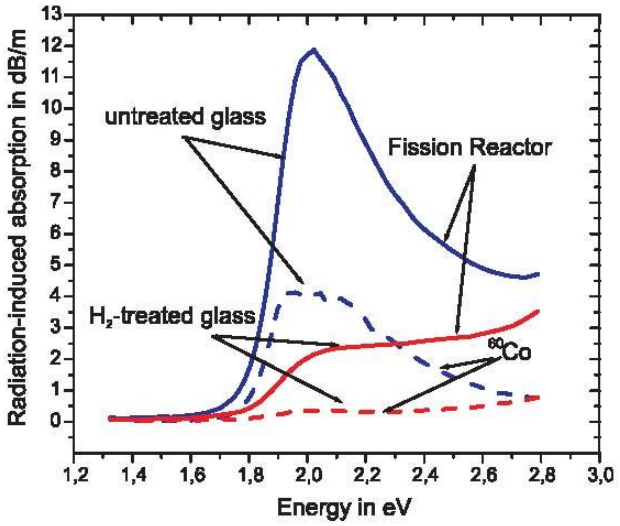
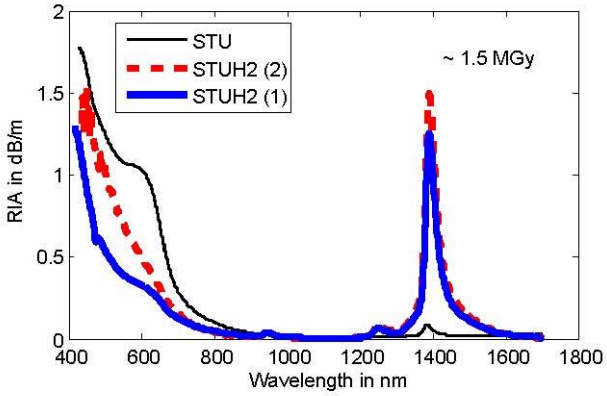
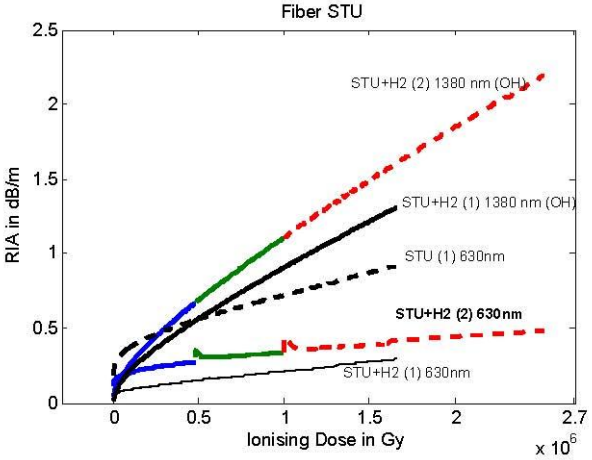


Fig 4: Spectral comparison of the RIA in the KU1 and KU1+H2 (KU2) fibres following a ^{60}Co irradiation up to 3 MGy or a fission reactor irradiation at the same ionising dose and about 2×10^{17} n/cm^2 (E> 0.1 MeV) [6].



(a) RIA spectrum at 1.5 MGy



(b) Dose Kinetic (1)= as received ; (2) two years later

Fig 5: RIA response in as-received STU fibre (marked (1) and curve from cf Fig. 11.4(c)) and irradiated two years later (marked as index (2)) in 3 successive tests (a) Spectral comparison at 1.5 MGy (b) Kinetic response with 3 consecutive irradiations (marked (2)).

ANNEX

INTERMEDIATE REPORT

SCK•CEN
STUDIECENTRUM VOOR KERNENERGIE
CENTRE D'ÉTUDE DE L'ÉNERGIE NUCLÉAIRE

Development and supply of large diameter, metal jacketed, hydrogen-loaded optical fibres
Task 05-1324 (Art 5.1b)

Deliverable EU-9.1 – First & Second Intermediate Report
6th August 2006

Benoît Brichard

1. Kick-Off Meeting

A kick-off meeting was held in Moscow begin February 2006 to discuss and agree on the specifications of the fibre fabrication and the hydrogen loading conditions. According to the agreed planning the fibres should be available end 2007 for irradiation testing at SCK•CEN.

It could be desirable, if necessary, to increase the number of targets for the irradiation assessment. As SCK•CEN has already in stock two 600 µm Al-coated fibres (low-OH and high OH), I suggested to send 50 meters of each fibre for hydrogen-loading. This length is required to install the fibre in the reactor section.

2. Determination of the hydrogen content

The impregnation by hydrogen of the silica network modifies the infrared optical spectrum by producing several hydrogen-induced absorption vibration bands at 1.245, 1.132 and 1.083 µm as depicted in Fig 1. The intensity of these bands reflects the hydrogen concentration. By measuring this intensity it is possible to estimate the hydrogen content if the extinction coefficient is known accordingly[1;2]. Hence it is recommended to monitor the evolution of the IR spectrum during the hydrogen loading process.

In the case of low-OH fibre, the hydrogen also reduces the drawing-induced band around 600 nm. It would be therefore also instructive to monitor this change if the available spectrometer has a sufficient spectral range.

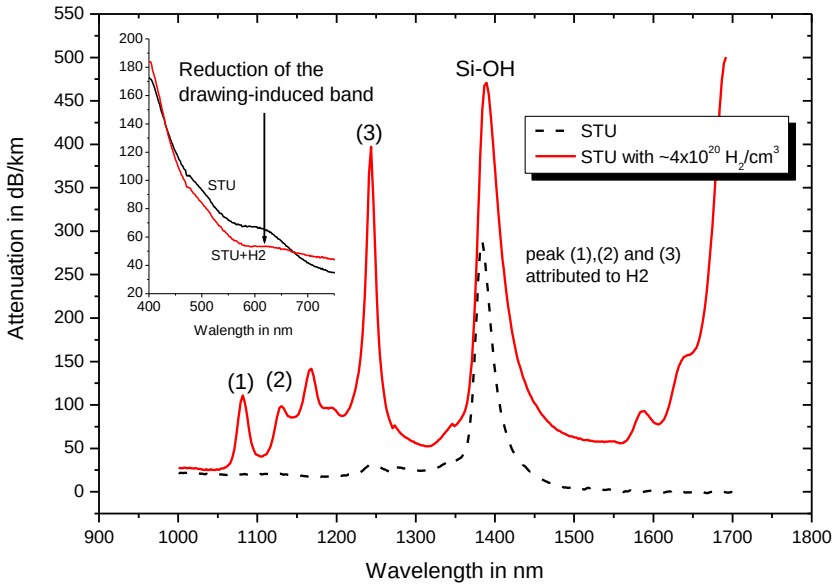


Fig. 1- Comparison of Infrared-optical attenuation spectra of H_2-treated and untreated low-OH unirradiated silica fibre. The inset shows the reduction of the drawing induced band due to the action of H_2.

3. First irradiation assessment of 600 µm in BR2

During the IRRCER task EFDA TW4-TPD/IRRCER Del 3[3] 200 µm and 600 µm Al-coated fibre was hydrogenated and irradiated in similar conditions in the BR2 reactor. Fig 2 shows a comparison of the RIA around 600 nm of a 200 µm fibre and a 600 µm fibre for a given irradiation dose. Clearly, the hydrogen loading was significantly much more efficient in the 200 µm compared to the 600 µm. To incorporate enough hydrogen into a thick fibre the exposure to hydrogen must be extended to several months at high temperature.

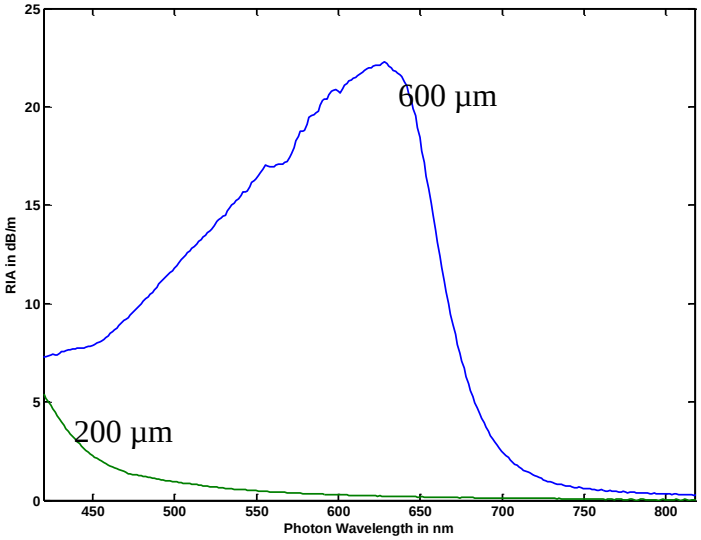


Fig 2: Comparison of the RIA in 600 µm SSU and 200 µm STU Al-coated pure silica core fibres loaded with hydrogen at the same time and irradiated simultaneously at 1.610^{16} n/cm^2 (E>0.1MeV) and 2 MGy.

The measurement of the RIA in thick fibre also appears more complicated because of the strong influence of the radio-luminescence. We can note indeed a distortion of the absorption spectrum in the 450-600 nm region. Fig 3 shows a comparison of the radio-luminescence in 200 µm and 600 µm fibres irradiated in BR2. Absorption measurement must be therefore corrected to take into account the luminescence effect.

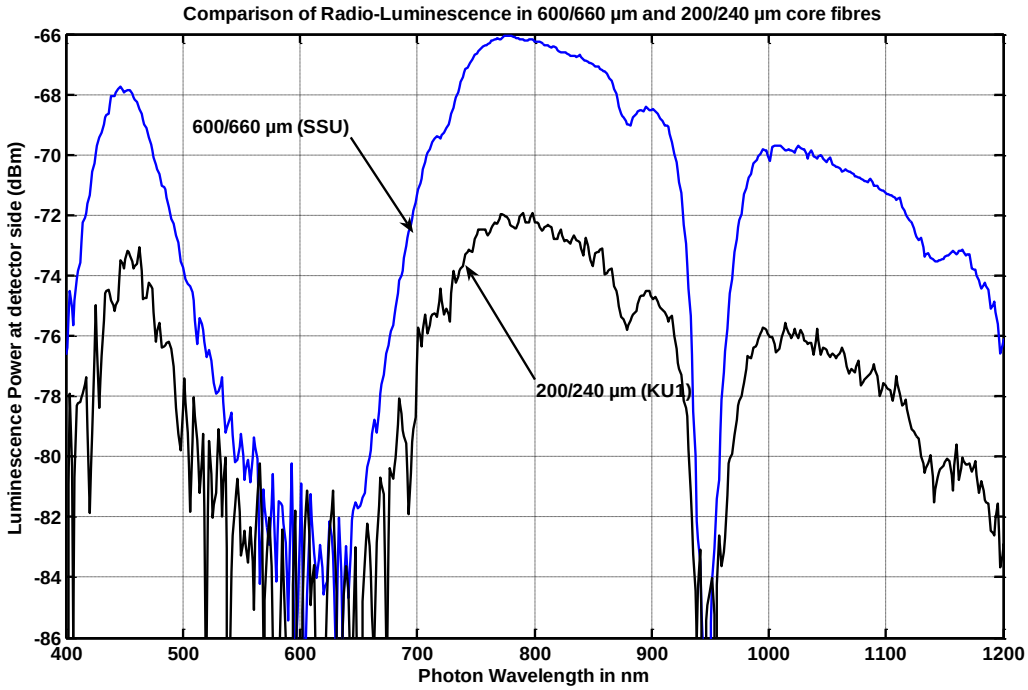


Fig 3: comparison of luminescence spectrum in Al-coated 600 µm SSU fibre and 200 µm KU-1 fibre.

An additional difficulty also arises with the radiation testing of thick fibres, which is the increase optical loss due to the bending (macro-bending loss). In the present test facility in BR2 (SMIRNOF) the useful diameter is 57 mm only which is too small for a proper testing of several 600 µm fibres. A large diameter irradiation facility (~150 mm) should be foreseen for such radiation test.

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