MEASUREMENT OF

WINDOW GLASS TEMPERATURE WITH A

FLUORESCENCE INTENSITY RATIO

OPTICAL FIBRE SENSOR

A thesis submitted

By

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For the degree of

MASTER OF SCIENCE

Optical Technology Research Laboratory

School of Electrical Engineering

Victoria University

2003

For JM, RC and BA.

Your word is a lamp to guide me

And a light for my path

Psalms 119:105

Declaration

I, Maria Cristina Vergara, declare that this thesis titled,

“Measurement of window glass temperature with a fluorescence intensity ratio optical fibre sensor”

Is my own work and has not been submitted previously, in whole or in part, in respect to any other academic award.

M. Cristina Vergara,

Dated the 12 December 2003.

Acknowledgments

There are many people to whom I am grateful for their involvement in the progression and fruition of this thesis. Principally, I wish to whole-heartedly thank Assoc. Prof. Gregory Baxter for his guidance, encouragement, persistence, and allowances of my other projects during the course of this work. The advice and support of Assoc. Prof. Stephen Collins have been invaluable and most appreciated. I also wish to thank Dr Scott Wade for always generously sharing his knowledge, and Dr Ian Mitchell for the meaningful discussions and encouragement.

Many thanks go to Dr Yaping He for the advice in regards to the fire aspect of this work. I am grateful to the Centre for Environmental Safety and Risk Engineering (CESARE) at Victoria University (VU) for the use of facilities and for the technical expertise of Mr Michael Culton during my time there. I give special mention to Ms Iraida Khanina. I am thankful for her friendship, and for her patience and accommodation of my work with her research.

I greatly appreciate the assistance of Hayrettin Arisoy, Nicoletta Dragomir, Donald Ermel (particularly for construction of the fibre holders), Peter Farrell, Bernhard Koziol, Abdurrahman Kuzucu, Thinh Nguyen, Maria Pylnyk, Alex Shelamoff, Sachin Surve, Corinna Sullivan, Jakub Szjaman, Robert Taylor, and other staff and students from the Applied Physics Section (VU). I have had important and interesting discussions with them as well as enjoyed their company.

I am grateful to Dr Gérard Monnom from the Laboratoire de Physique de la Matière Condensée, Universitè de Nice, France, for the doped fibres. I also wish to acknowledge the provision of a scholarship from the School of Communications and Informatics (now Electrical Engineering), VU.

Thank you to Bernadette Palmer and other family and friends, especially my parents José and Rustica Vergara, for your support and love, from start to finish.

I cannot express enough my thanks to my best friend (and coach) Bryan Sharp. Thank you for your love, faith, and the endless cups of tea. To Chi Chi and Johnny, thank you for your resilience, and for the giggles and dancing along the way.

Abstract

A prototype optical fibre temperature sensor employing the fluorescence intensity ratio (FIR) technique has been utilised to measure the surface temperature of window glass during a fire. A comparison has been made between the intrinsic optical fibre sensor and thermocouples (the conventional temperature measuring device used in fire research).

Neodymium doped fibre was used as the temperature sensing element. Due to the destructive nature of the fire tests on the neodymium doped fibre, a new section of bare doped fibre was spliced for each test. Before each fire test, the (non-glass) coating on the sensing fibre was removed. The detector was calibrated against K-type thermocouples in a stabilised temperature environment. This same bare optical fibre was attached to the window glass pane with an adhesive that was appropriate for the fire test conditions.

A preliminary investigation was undertaken to compare the effect of a radiant heat environment on the sensing fibre and thermocouples. The bare fibre showed a significantly lower temperature (about 25°C lower) than the average thermocouple temperature. This is consistent with the expectation that the fibre is less subject to the effects of radiation than the thermocouples. Thus it was demonstrated that the thermocouple did not accurately represent the time-dependent temperature behaviour of window glass, and specifically, the temperature within the core of the fibre.

Fire tests were conducted in a chamber, which was designed and built to simulate a combustion fire starting in a small room with a window. Both sensor types were used to measure the temperature of the inside surface of the window glass during a fire. The observed temperature difference was smaller than that observed during the preliminary tests where the thermocouples were subject to a radiant heat environment. Reasons for this are discussed within the thesis.

The measurement of window glass temperature during a fire has been demonstrated using an optical fibre sensor based on the fluorescence intensity ratio technique. This clearly establishes the existence of a discrepancy between window glass temperature and the temperature measured by thermocouples when they are subjected to a radiant heat environment.

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Chapter 1

Introduction

1.1 OPTICAL FIBRE SENSORS

The development of optical fibres for telecommunications has resulted in many technological advances that have enabled the integration of optical fibres in, and as, sensors. The attractions of optical fibres in telecommunications such as: low susceptibility to electromagnetic interference, small size and weight, low transmission loss, and their capacity to operate in hazardous environments are well known. These advantages of optical fibres are pertinent for their use in temperature sensing [Dakin and Culshaw, 1988].

The basic operation of an optical fibre sensor system is illustrated by Figure 1.1.1. The sensor usually consists of an appropriate optical source, which is guided through fibre onto a sensor placed in the environment to be measured. The measurand (temperature, displacement, or strain, etc.) alters one or more optical parameters of the light (such as for example: intensity, phase, amplitude.) A returning fibre guides this modified, counter-propagating light to an optical detector. This signal is then appropriately processed to provide the value of the measurand.

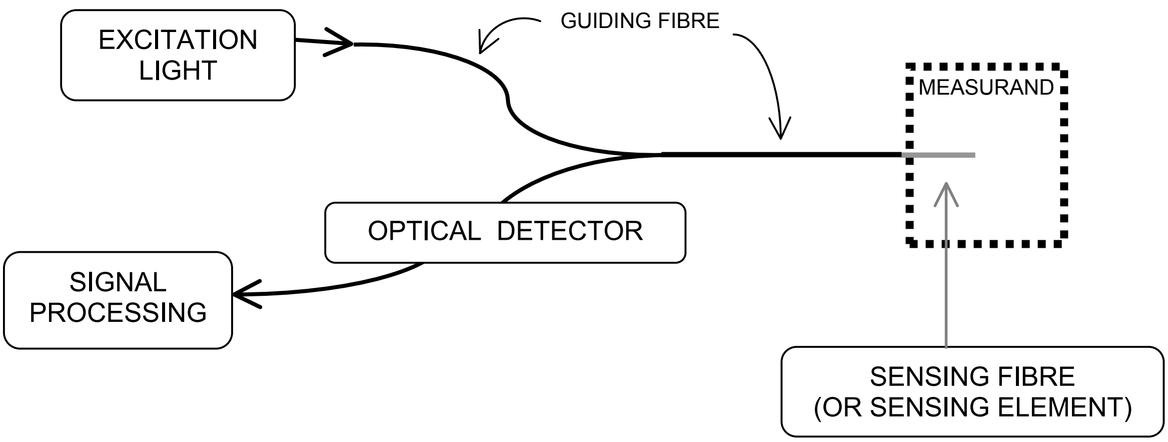


Figure 1.1.1 the basic operation of an optical fibre sensor system.

Optical fibre sensors can be categorised in many ways, the most general classification being either intrinsic or extrinsic. Intrinsic fibre sensors are those in which a section of fibre is the actual sensing element. Extrinsic fibre sensors are those where the purpose of the fibre is to simply transmit the optical signal to and from a non-fibre modulator. In this work, the sensor used is intrinsic. Optical fibre with neodymium ions doped into its core acts as the sensing element. Further description of this sensor is provided in Chapter 2.

1.2 OPTICAL FIBRE TEMPERATURE SENSORS

There are many temperature dependent optical properties on which optical fibre temperature sensors are based. The required application of a temperature sensor will often determine one type being chosen over another. For example, if a relatively fast temperature measurement at a single location is desired, a point temperature sensor is suitable. For temperature monitoring of large or long structures such as tunnels or pipelines, a distributed temperature sensor is used, although the time of processing is normally slower. Other attributes of a temperature sensor, such as its temperature range, accuracy and cost, will also determine its applicability. A short review on well-regarded absorption and fluorescence based techniques is presented here.

1.2.1 Absorption Based Temperature Sensors

Some temperature sensing schemes are based on the temperature dependent optical absorption of a material. Two such sensing materials used are semiconductors and rare earth doped fibre.

An early extrinsic optical fibre sensor by Kyuma *et al* [1982] used a semiconductor material as the sensing element. A section of gallium arsenide was attached to the end of an optical fibre. Near ambient temperatures (300 K) the energy band gap of such materials is almost inversely proportional to temperature. That is, the wavelength relevant to the fundamental absorption edge moves toward longer wavelengths with increasing temperature. This is illustrated in Figure 1.2.1.

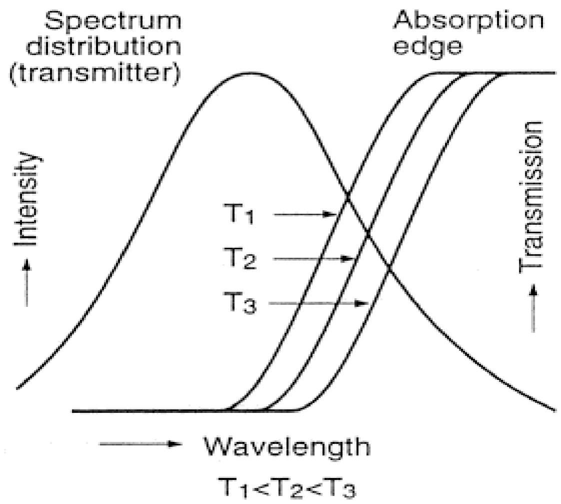


Figure 1.2.1 Semiconductor transmission as a function of wavelength and temperature [Kyuma *et al,* 1982].

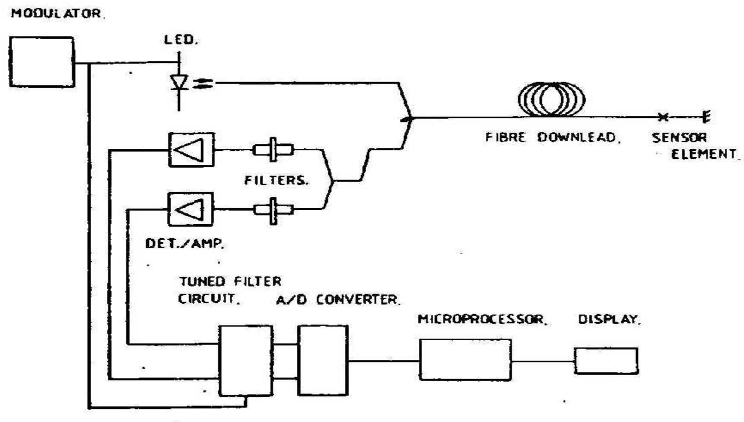
The optical temperature measurement system used two light emitting diodes of different wavelengths (λ_1 and λ_2). The sensor's absorption of light at one wavelength (λ_1) changed as a function of temperature. The light of the second wavelength (λ_2) was used as a reference. An accuracy of ±1 °C was achieved for the temperature range of -10 to 300 °C. The sensor had a response time of 2 seconds.

Rare earth doped fibres, namely neodymium doped fibres, have been used in temperature sensing schemes by different groups of workers [Appleyard *et al,* 1990;

Morey *et al,* 1983; Voger, *et al,* 1993]. The schemes utilised the temperature sensitive absorption properties of the fibre. The ratio of absorption intensities at two separated wavelengths was calculated to give the temperature.

A schematic of the self-referencing system used by Appleyard *et al* [1990] and the change in absorption with respect to temperature relative to 20 °C are shown in Figure 1.2.2 (a) and (b) respectively. The sensing element used was 25 mm in length. An accuracy of ±1 °C was achieved for the 20 to 200 °C range of temperatures.

(a)



(b)

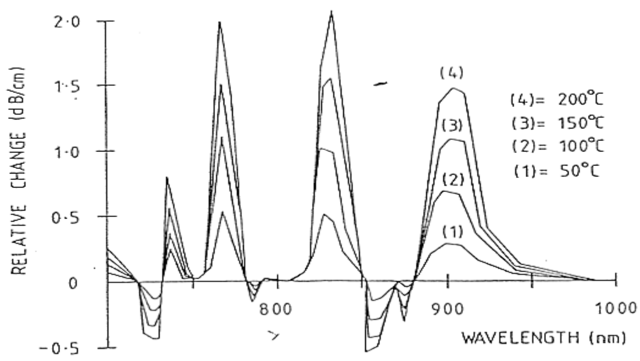


Figure 1.2.2 (a) Schematic diagram of the sensor system (b) The change in absorption with respect to temperature (relative to 20°C) by Appleyard *et al* [1990].

If absolute absorption intensity at only a single wavelength is measured to provide the temperature, signal intensity fluctuations and connector losses can give inaccurate temperature readings. The differential absorption technique overcomes these problems (which are not necessarily temperature related), since the ratio of absorption intensity at two separated wavelengths is calculated to provide the temperature information.

1.2.2 Fluorescence Based Temperature Sensors

The temperature dependent fluorescence from particular materials is used in several sensing schemes. Usually, the emitted fluorescence intensity from the material is used directly, or the decay time of fluorescence is calculated to monitor the temperature.

One early commercially available temperature sensor was the ASEA 1010 model [Ovren *et al*, 1983], with an LED source illuminating a gallium arsenide sensing element uses the ratio of emitting fluorescence to measure temperature. Taking the ratio of fluorescence at two wavelengths overcomes inaccurate measurements, which may arise due to fluctuations in the excitation source. Currently available are the Luxtron m600 and m3300 Series Fluoroptic® Thermometers. Wickersheim and Alves [1982] reported an early commercial version of these sensors. The current sensors measure the fluorescence decay time from a phosphorescent material that acts as the sensing element. This phosphorescent material is fixed to a length of optical fibre. The variation in decay time is proportional to the change in temperature. The accuracy of this system is 0.2 °C [Luxtron, 2004].

Different groups have measured the fluorescence decay of different materials to monitor temperature [Sholes and Small, 1980; Wickersheim and Sun, 1987; Grattan *et* *al,* 1988; Fernicola and Crovini, 1995]. A commercial sensor utilising this temperature dependent fluorescence decay time is the Luxtron 750 fluoroptic model (a magnesium fluorogermanate being the phosphor) [Wickersheim, 1987]. In general, a pulsed light source is required to excite the fluorescent material and an exponential decay signal is emitted. The decay time, τ, is indicative of the temperature.

The most direct (and early) method of analysis of the decay [Grattan and Palmer, 1984; Wickersheim and Sun, 1987] is illustrated by Figure 1.2.3. Firstly, the decaying signal, I_o, (at a fixed time, t_1,) is measured. The time, t_2, is recorded when the decaying signal falls below a calculated reference level (I_o/e). The time constant, τ, of the exponential decay signal is the interval between t_1 and t_2. Other signal analysis methods, which give a more precise calculation of lifetime, have been used. Grattan and Zhang [1995] have reviewed further details of these.

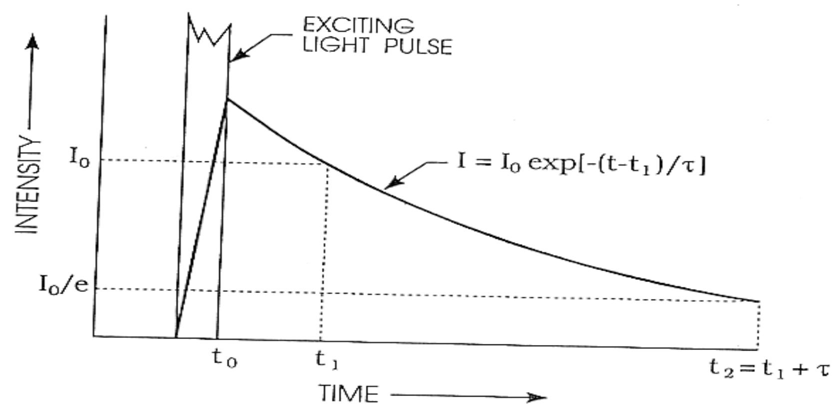


Figure 1.2.3 The two-point constant measurement is one technique used to measure fluorescence decay [Wickersheim and Sun, 1987].

Researchers using the fluorescence lifetime method found that, subjecting the sensing probe to an elevated temperature for an extended period of time has a significant impact on the performance of the sensor [Zhang *et al*, 1998]. The effects of annealing neodymium doped fibre used in fluorescence lifetime temperature measurements were reported. It was found that exposing the neodymium doped fibre to a temperature of about 750 ºC for a suggested minimum of 100 hours stabilised its fluorescence lifetime value. A small sensor probe (of about 5 mm) capable of operating at a maximum of 750 ºC was demonstrated. Further work on this fluorescence lifetime system raised the upper operating temperature to 1100 ºC by the use of erbium doped fibre as the sensing probe [Zhang *et al*, 1998a]. The erbium doped fibre underwent annealing at 1100 ºC for around 40 hours, after which, the lifetime stabilised. The fibre was subjected to this temperature for a further 100 hours and yielded stabilised lifetime values. This treatment of the sensing material seems to be an important one as it potentially increases stability and the maximum operating temperature of the sensor. The effects of annealing other rare earth doped materials or fibres, used in fluorescence decay temperature studies, have been reported [Sun *et al*, 1998 and 2000; Zhang *et al*, 2000].

Annealing effects have also been studied in fluorescence intensity ratio temperature measurement systems. The fluorescence intensity ratio technique is used in this body of research. This technique involves measuring the fluorescence levels from rare earth doped optical fibre. A comprehensive review of the work using fluorescence intensity ratio systems is presented in Chapter 2.

1.2.3 Other Sensing Techniques

Blackbody Radiation and Pyrometry

A well known commercial optical fibre sensor, which measures the blackbody radiation of a material to give temperature, is a system designed by Accufiber [Accufiber Inc., 1988]. The Accufiber model 100 consists of a sapphire rod tipped with a thin metal film. The top of the probe forms a blackbody cavity. The radiance emitted from the cavity is determined by Planck's equation (which describes black body radiation) in conjunction with Gouffe's equation (which describes finite-dimension cavity emission) [Dils, 1983; Cooper, 1985]. The temperature is measured from the narrow wavelength band radiance emitted from the cavity (Figure 1.2.4). The sapphire rod and the guiding fibre transmit the radiation to an optical detector. The operating temperature range of the sensor is 300 to 2000 °C with an accuracy of 0.10 % at 1000 °C. The sensor is most practicable at temperatures above 500 °C [Grattan and Zhang, 1995].

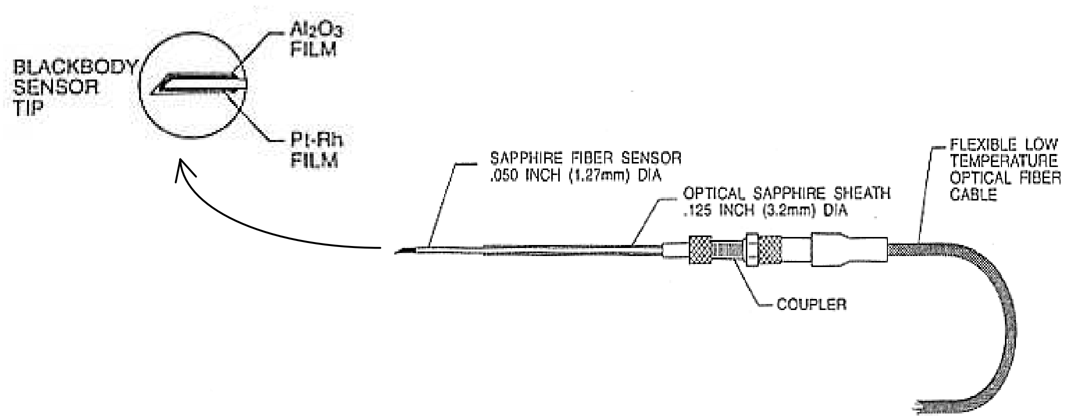


Figure 1.2.4 A black body radiation sensor [Accufiber Inc., 1988].

In addition to being a remote temperature sensing method, optical pyrometry is essentially based on the same principles of temperature using black body radiation as described in the technique above, but with the surface being measured not touching the actual sensor. The sensor is particularly useful for high temperature environments or for when in-situ sensing is not feasible. Similarly a sapphire sensor may be coupled to an optical fibre, which transmits the radiation to a light detector. The Accufiber Models 10 and 100 are black body radiation sensors (which can be manufactured for contact or non-contact specifications) and are available commercially by the Luxtron Corporation [Luxtron, 2004a]. Although, these sensors are most reliable for very high temperature applications (maximum of 1600 °C), are able to withstand harsh environments, and have very fast response times, they are unsuited for the particular work of this thesis. This is because of the complication of distinguishing the radiation emitted from the surface of window glass with the substantial surrounding radiation present due to the fire.

Interferometry

Optical fibre interferometers are based on changes in the phase or polarisation of light. A change in temperature will result in a phase difference between the modulated light in the measuring arm and light in the reference arm (which is unaffected by the measurand). The schematic of one such commercially available sensor using Fabry-Perot interferometry to measure temperature is shown in Figure 1.2.5. This sensor, by Saaski and Hartl [1992], consists of a Fabry-Perot etalon and is based on two-wavelength ratio calculations, which help to eliminate intensity fluctuations not related to temperature. Earlier interferometric sensors have been described [Corke *et* *al*, 1985; Akhavan Leilabady *et al*, 1986]. Although these systems have high resolution they are not absolute devices and require calibration [Dakin and Culshaw, 1989], making them more useful for measuring temperature differences rather than actual temperature. If laser sources are required to excite them, these further add to the cost of such systems.

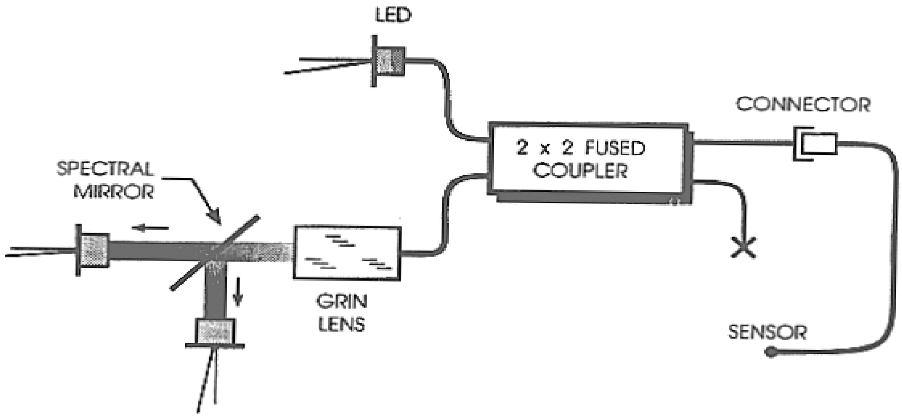


Figure 1.2.5 A Fabry-Perot interferometric sensor [Saaski and Hartl, 1992].

Distributed Temperature Sensing

Some situations may require the use of a distributed temperature sensor; that is a sensor consisting of a single fibre, which can monitor temperature continuously at points along a required length. As examples, Raman and Brillouin scattering have been utilised for distributed sensing. Basically, the scattering mechanisms occur due to light interacting with the molecules of the fibre, causing shifts in the input wavelength. Calculating the ratio of the intensity of the modulated light at these wavelengths gives the temperature [Grattan, 1987]. Optical time domain reflectometry is used to measure the scattering. Both York and Hitachi have commercial distributed temperature sensors based on laser induced Raman scattering. Such sensors have a long data collection time (typically of the order of minutes), making them unsuitable for fire experiments due to the quickly changing conditions of a fire.

Thermal Imaging

Thermal imaging is the measuring of the distribution of infrared radiation emitted from the surface of an object. This measured thermal radiation is then converted to an image that can be displayed. Thermal imaging systems usually include an optical configuration, a detector, processing electronics and a display [Childs, 2001]. Although thermal imaging is not an optical fibre based temperature sensing scheme, it has been included here as the use of thermal images is becoming more widespread and it is often utilised in fire research. Thermal imagers are being used in commercial, industrial, military and medical applications. The thermal images are appealing because they are remote, non-invasive, and they do not require an illumination source. Further, they provide quantitative, and mostly qualitative information, in a high quality visual form. Thermal imagers, however, are relatively expensive. Thermal imaging is not a viable method for the fire research undertaken in this thesis because, as with black body radiation sensors, thermal imaging depends on the detection of radiation from a surface. It would be difficult to discriminate the temperature of the windows from the significant background radiation present in the combustion environment.

Comprehensive reviews of the operating principles of these and other optical fibre temperature sensing techniques are contained in the literature [Grattan and Zhang, 1995; Dakin and Culshaw, 1989; Grattan, 1987; Hartog, 1987]. There are numerous available and suggested techniques to measure temperature, with each method having its attractions and limitations. A comparison of techniques have been made analytically and experimentally by other groups [Sun *et al*, 1999; Berghmans *et al*, 1998; Keck, 1996], thus illustrating that temperature sensors are now being considered for more novel applications. This emphasises the importance of, and continuing interest in, temperature sensor development.

1.3 OPTICAL FIBRE STRAIN AND TEMPERATURE SENSORS

1.3.1 Cross Sensitivity of Optical Fibre Strain Sensors with Temperature

The topic of cross sensitivity of fibre temperature sensors with strain is raised here, as concurrent temperature and strain fibre sensing is an area gaining considerable interest. It is also a significant concern in this work of window glass temperature measurement. This is because during the fire tests, the window glass may experience strain. It is important that any effects of strain are not mistakenly interpreted as temperature effects.

Generally, optical fibre sensors that measure strain are also characteristically sensitive to temperature. Accordingly, a particular compensation configuration may be required to ensure that the effects due to temperature are not erroneously measured as changes in strain. Conversely, a sensor whose objective is to measure temperature alone may be adversely impacted by the corollary of cross sensitivity to strain. An extra compensation arrangement may be needed. Additional complexities to the temperature sensor invariably invoke a rise in its cost. Thus, in design and monetary terms, it is highly desirable to have a fibre temperature sensor, which is independent of strain effects.

The fluorescence intensity ratio technique has been found to have a strain sensitivity that is practically zero [Wade *et al*, 2001], making it suitable for this work of window temperature sensing during fires. Further, instead of precluding the technique from use in a dual temperature and strain sensor, this non-sensitivity to strain has meant that the fluorescence ratio technique is also highly desirable for use in a dual temperature and strain fibre sensor when placed in conjunction with another sensor for strain [Collins *et al*, 2002]. Work in this particular area is reviewed in Chapter 2.

1.3.2 Dual Strain and Temperature Fibre Sensors

The measurement of temperature and strain at the same time is useful for a number of applications such as the continual assessment of civil and industrial structures. One sensor to measure the two parameters is preferable to the use of two sensors. The use of two sensors means more sophisticated design and expense. Also, the system may not be capable of measuring at the same location. Single systems that have demonstrated simultaneous measurement of strain and temperature have been reviewed [Jones, 1997; Jin *et al*, 1997]. Those comprising of intrinsic fibre systems are generally less costly than those using other optical methods. Further, they can be used in electromagnetically prone environments. Some of the most prominent dual measurement systems reviewed [Rao *et al*, 1998] are those using a fibre Bragg grating in conjunction, for example, with another fibre Bragg grating or other optical measuring technique [Xu *et al*, 1994; Ferreira *et al*, 1996].

Fibre Bragg grating sensors are mainly used to measure strain and may additionally be exploited to measure temperature. Radiation within the fibre core, of wavelength λ_B, will be reflected by a Bragg grating, when:

$$\lambda_B = 2mn_1\Lambda,$$

where m is an integer, Λ is the periodicity of the grating, and n_1 is the refractive index of the core. Changes in strain will affect both the grating spacing and the refractive index of the core. Temperature affects both the grating wavelength and the refractive index of the core. The advantages of fibre Bragg grating sensors are their relatively high resolution of temperature and strain (however, their inherent strain sensing capabilities are inappropriate for use in the fire experiments of this work). Additional benefits are that the output is wavelength-encoded and linearly related to the measurand, and that they are relatively easy to construct [Wilson and Hawkes, 1998].

Temperature and strain effects using different fibre Bragg grating techniques have been reported [Morey *et al*, 1992; Xu *et al*, 1994; Ferreira *et al*, 1998]. Sensors using a combination of fluorescence and fibre Bragg grating methods are advantageous. This is because of the possibility of using one excitation source and detection system for strain and temperature measurements, and the convenience of a sensing fibre to reduce costs, since other simultaneous temperature-strain measuring schemes are relatively expensive to implement [Sun *et al*, 1999].

Work by Trpkovski *et al* [2003] regarding a dual temperature and strain system utilising the fluorescence intensity ratio method and a fibre Bragg grating, is discussed in Chapter 2. A dual temperature and strain sensor employing fluorescence lifetime decay and a fibre Bragg grating technique was demonstrated by Wade *et al* [2001a]. The standard deviation errors from the calibration results were 20.4 $\mu\varepsilon$ for a strain range of 22-1860 $\mu\varepsilon$ and 1.2 ºC for a 25-120 ºC temperature span. The sensing section consisted of erbium doped fibre spliced to a fibre Bragg grating (with a centre wavelength of about 1548 nm); the total sensing length being about 8 cm. Figure 1.3.1 illustrates the experimental arrangement used for calibration in this work. (The sensor is labelled sensor 1 in the inset.) The sensor was based on the measurement of the fluorescence lifetime of the erbium doped fibre and the Bragg wavelength shift. The generated fluorescence from the erbium doped fibre was also used as the interrogation source for the fibre Bragg grating. This eliminated any additional laser requirements. Furthermore, it did not affect the measurement of the fluorescence lifetime changes, which is a significant consideration for temperature correction.

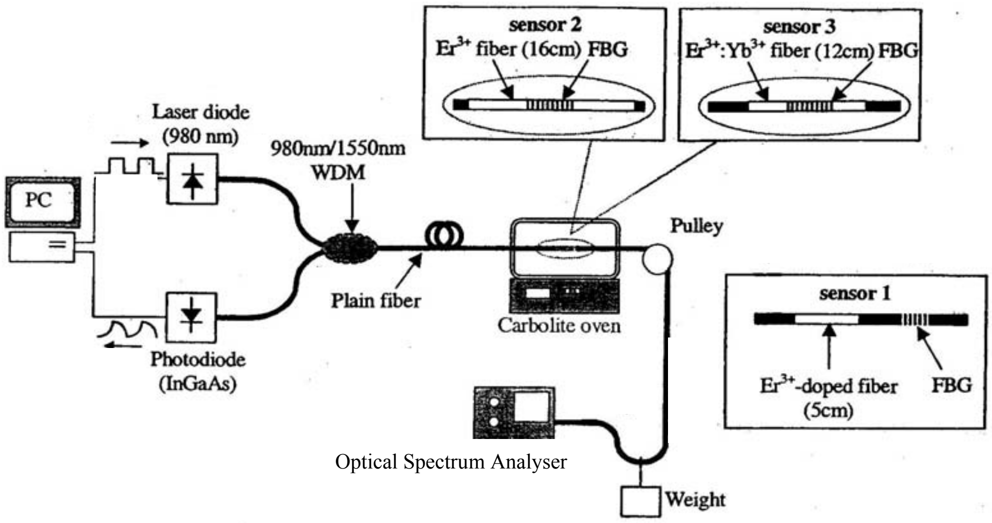


Figure 1.3.1 Experimental arrangement used for the calibration of the dual temperature and strain sensors. The doped fibre and the fibre Bragg grating (FBG) configurations are shown. Sensor 1 is that used by Wade *et al* [2001a]. Sensors 2 and 3 are those used by Forsyth *et al* [2002].

The continuation of this work was reported by Forsyth *et al* [2002]. The experimental arrangement used for calibration of the sensors is the same as for that described by Wade *et al* [2001a]. Two different sensors were constructed where the fibre Bragg grating was written into doped fibre, facilitating the coincident location of the temperature and strain sensors. One of the sensors (labelled as sensor 2 in the inset in Figure 1.3.1) was a section of erbium doped fibre (of length 16 cm.) A fibre Bragg grating, at the particularly chosen centre wavelength of 1551.5 nm, was written into the single ion-doped fibre. The other sensor (depicted as sensor 2 in the inset in Figure 1.3.1) comprised single mode fibre co-doped with erbium and ytterbium ions. A fibre Bragg grating (of 1549.7 nm centre wavelength) was written into the 12 cm length of fibre. The three sensors showed essentially the same strain sensitivities. The standard deviation error for strain was 7 με (sensor 3) for a strain range of maximum 1860 με. For temperature, the standard deviation errors were 0.8 ºC (for sensor 3) and 1.5 ºC (for sensor 3), for a range of temperatures to 120 ºC. The results from these studies illustrate the potential of this simple yet effective sensing scheme.

1.4 MEASUREMENT OF WINDOW GLASS DURING FIRES

Domestic fires can result in the destruction of property, which have the potential for extreme financial ramifications. More tragically, fires sometimes result in the loss of lives. In order to prevent these deaths and limit the amount of property damage, models of fire performance are used in the development of building safety standards. Trying to predict the behaviour of fire is difficult because of its chemical and physical complexity. An important factor in the performance of fire is the role of window glass.

The function of windows is considered to be of significance in fire modelling. In a compartment fire, the environmental conditions are continuously changing. It is difficult to formulate mathematical equations to describe the processes occurring during a fire, although this has been done with some success [He and Beck, 1995; Cuzzillo and Pagni, 1998; Joshi and Pagni, 1994 and 1994a]. Windows can hinder the spread of fire by acting as barriers if they remain intact. Conversely, if they break and fall out they become a major ventilation source, facilitating the fire growth along a building [Emmons, 1986; Joshi and Pagni, 1994]. Therefore, predicting the moment at which glass breaks is of vital consequence [Cuzzillo and Pagni, 1998; Hassani *et al*, 1995].

In domestic fires, the uneven heating of different sections of window glass directly by the fire, induces tensile stress on the glass causing it to break [Emmons, 1986]. This breakage occurs at a critical temperature; the temperature difference between the middle portion of the pane and the glass edge (which is usually fixed and remains at a lower temperature). Independent workers proposed theoretical models with critical temperatures of about 58 °C [Pagni, 1988] and 80 °C [Keski-Rahkonen, 1988]. The disparity in these critical temperatures is due to the assumption of different material constants. Subsequent experiments found the critical temperature to be up to 90 °C [Silcock and Shields, 1993; Skelly *et al,* 1991; Joshi and Pagni, 1994a]. The inconsistency in the theoretical and experimental values was attributed to radiative heating of the thermocouple. It was concluded that the measured value was higher than the actual temperature of the glass, so that the experiments could be interpreted as concurring with the theoretical proposals. Thus difficulties arise with distinguishing the genuine temperature from the effect of radiation [Janssens and Tran, 1992] due to the use of conventional thermocouples for window glass measurements. A thermocouple does not truly represent the surface temperature of window glass in a fire because of discrepancies in thermal properties [Luo, 1997; Hassani *et al*, 1995].

For fire experiments of window glass, the use of a glass sensor, with similar optical properties to windows, is desirable to overcome problems associated with thermocouples. As the optical fibre is in effect transparent, the disturbance to the temperature environment by the fibre would be minimal. For this research, commercial fibre sensors are unsuitable, as they are expensive and the fire tests are of a destructive nature. Temperature sensors based on the detection of thermal radiation are also unsuitable, due to the complexity of differentiating the radiation emitted from the glass and the presence of radiation due to the fire. Further, the sensing element is usually encased in a material dissimilar to glass. It is therefore desirable to employ a sensor where a new section of sensing fibre can be readily and inexpensively spliced for each fire test. Due to the dynamic nature of fire, a point temperature sensor is required which has relatively high resolution and fast response time. Significantly, the coating on the sensing fibre needs to be removed to more genuinely match the response of window glass to heat radiation in a fire. In this work, a temperature sensor based on the fluorescence intensity ratio technique was used in conjunction with thermocouples. For measurement of the window glass temperatures during the fire tests, the typical measurement range required was from ambient temperatures to about 200 ºC. As is discussed in Chapter 2, the fluorescence intensity ratio technique has the advantage of being practically insensitive to strain, an important consideration for glass in the fire environment.

1.5 AIMS OF THIS RESEARCH

The purpose of this research was to employ an optical fibre temperature sensor for the measurement of window glass temperature during fires. This was part of a collaborative investigation into the mechanism of window glass breakage in building fires by the Optical Technology Research Laboratory (OTRL) and the Centre for Environmental Safety and Risk Engineering of Victoria University. An existing neodymium doped sensor based on the fluorescence intensity ratio technique was used in conjunction with thermocouples to measure the temperature. Specifically, the optical fibre sensor collected data on the temperature of window glass in small scale and large scale fire tests. The effect of radiant heat on thermocouples (the conventional temperature sensing devices in fire research) was quantified.

This work contributes new knowledge to two areas: fire research and optical fibre temperature sensing. A better understanding of the behaviour and function of window glass breakage during fires was obtained. An existing prototype neodymium doped sensor based on the fluorescence intensity ratio technique was used for the fire simulation tests. The reliability and applicability of the sensor were demonstrated. The use of the sensor for this novel application verifies the suitability and operating capability of the technique in a difficult environment. The compartment fire environment was hazardous, affected by strain (the thermal expansion of glass), and produced radiant heat. This research, using the fluorescence intensity ratio technique for the measurement of the thermal response of window glass in fires, is the first of its kind known to the author. The conclusions will prove helpful in the consideration of other fluorescence based methods to be applied to further work in fire research.

1.6 SCOPE OF THE THESIS

This thesis contains five chapters. The first chapter outlines the motivation for this research and is an introduction to optical fibre temperature sensing techniques. It aims to place into context the use of the fluorescence intensity ratio sensor (for investigations with window glass during fires) with other temperature sensing schemes. Chapter 2 is a review specifically on optical fibre temperature sensors based on the fluorescence intensity ratio technique. Its development as an important sensing technique and its applications are presented. Chapter 3 gives more detailed aspects, namely the characteristics of neodymium doped fibres for the fluorescence intensity ratio technique. The experimental investigation of this work is presented in Chapter 4. Details of the application of the sensor to the experiments of window glass during fires, and the results of these fire tests are included. Finally, overall conclusions relevant to the aims of this research are presented in Chapter 5.

Chapter 2

Temperature Sensing with the Fluorescence Intensity Ratio Technique

2.1 INTRODUCTION

In addition to the recognised benefits of optical fibre sensors, they have particular advantages (such as high immunity to electromagnetic interference) over other temperature measuring systems, for example, thermocouples. However, in order for a sensor to gain wide acceptance and commercial viability it also needs to meet further requirements. Some of these requirements may include reasonable price, straightforward operation, accuracy and reliability, measurement of a broad temperature range, and demonstrated, practical use in specific applications.

A temperature sensor with the capacity of the above may be achieved by consideration of factors such as the laser source and the sensing material. For fluorescence intensity ratio sensing, the sensing material is the fibre host and the dopant rare earth ion. They can, for example, affect the sensitivity of the sensor. They must be chosen well to achieve the best possible performance of the sensor and to minimise the cost. The realisation of a commercial temperature sensor based on the fluorescence intensity ratio temperature sensing method is likely. It has been demonstrated that the fluorescence intensity ratio technique has the potential to achieve the desirable qualities of a commercial device.

The fluorescence intensity ratio (FIR) scheme has proven to be an important and relevant one. Its suitability for use in this work, that is the focus of this thesis, of measuring the temperature of window glass during fires in conjunction with thermocouples, exceeds that of alternative types of sensing methods available. The fluorescence intensity ratio technique has been shown to have many attractive qualities. The system can be illuminated by relatively cheap excitation sources (such as laser diodes); the optical arrangement and detection system is relatively simple, yet effective; and it is insensitive to source intensity fluctuations and strain. The early work of the FIR technique is reviewed and its development as a significant sensing system is presented here.

2.2 PRINCIPLES OF THE FLUORESCENCE INTENSITY RATIO TECHNIQUE

In 1996, Baxter *et al* [1996] reported comprehensive details of an optical fibre temperature sensor based on the fluorescence intensity ratio technique. This early work demonstrated the advantages and some applications of the FIR technique and provided the basis to undertake further work in constructing a viable temperature sensor.

The FIR method is dependent upon the relative population of two thermally linked, excited state energy levels in a rare earth ion. The populations of the two excited levels are dependent on temperature. The radiative decay from these two levels to a common lower level results in two adjacent bands in the fluorescence spectrum. The temperature of the rare earth material can be established from measuring the amount of fluorescence from each level with the use of suitable filters. The ratio of the fluorescence intensity of these two levels is related to temperature by a Boltzmann distribution.

Figure 2.2.1 illustrates a simplified energy level diagram for a generic rare earth ion suitable for use in a FIR sensing scheme. The rare earth ion absorbs light of an appropriate wavelength, exciting it into the pump band. From this pump band it populates one of the coupled excited energy levels being considered (labelled 2 and 3 in Figure 2.2.1). Due to the large number of rare earth ions that are similarly excited, the relative population of thermalising levels may be predicted via a Boltzmann distribution [Koechner, 1996; Svelto, 1998]. The number of radiative transitions from each of the excited levels to a common lower level (1 in Figure 2.2.1) will follow the excited state populations and emission cross sections. The rare earth ions will then decay quickly to the ground state.

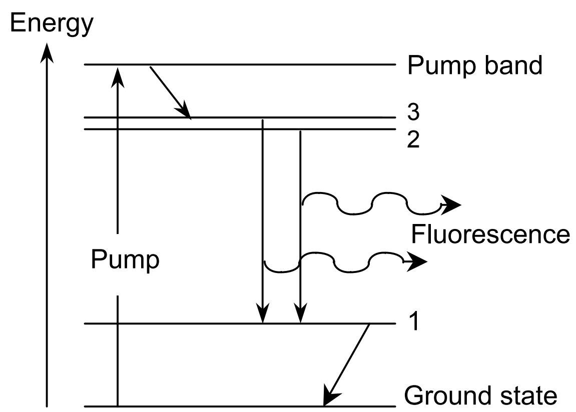


Figure 2.2.1 Simplified energy level diagram for a generic rare earth ion showing fluorescence properties [Baxter *et al,* 1996].

The FIR, labelled *R* for convenience, is given as:

$$R = \frac{\sigma_{31}(2j_3 + 1)}{\sigma_{21}(2j_2 + 1)} exp \left[\frac{-\Delta E}{kT} \right], \qquad (1)$$

where σ is the emission cross section of each of the excited state levels to the lower energy level, (2j+1) is the degeneracy of the given level, ΔE is the energy gap between the two thermalising levels, *k* is Boltzmann's constant, and T is temperature in kelvin.

The advantages of employing the strongly coupled levels, which thermally follow a Boltzmann distribution have been detailed by Wade *et al* [2003] and are briefly related here. The behaviour of the relative changes in the amount of fluorescence from the closely spaced thermalising levels is easily accounted for since the background theory is reasonably straightforward. The reliability of the sensor is enhanced by its independence of changes in the excitation pump. This is because the number of ions in each single thermalisation level is in direct proportion to the total number of ions. Fluctuations in the excitation source will equivalently impact on the fluorescence intensity from individual energy levels. Consequently, the excitation source changes will not induce an overall effect. Wavelength effects caused by bends in the fibre are minimised by the choice of closely spaced energy levels (about 1000 cm^{-1} or less), which will ensure that the fluorescence wavelengths being measured are near to each other.

An important feature is the rate of change of the fluorescence intensity ratio with the temperature, as a means to characterise the accuracy of the sensor.

The temperature sensitivity, S, for a generic system is given by:

$$S = \frac{dR}{dT} \cdot \frac{1}{R} = \frac{\Delta E}{kT^{2}}. \qquad (2)$$

Figure 2.2.2 shows how FIR and temperature sensitivity varies with the energy gap between the two thermalising levels, at room temperature, for a number of rare earth ions as discussed by Baxter *et al* [1996]. Generally, the choice of a larger energy spacing will improve the sensitivity of a sensor. However, the risk of too wide a gap is that the pair of energy levels may no longer be related by a Boltzmann ratio. Practically, this translates as a small fluorescence intensity level in one detector. Consequently, signal to noise difficulties may be incurred due to low fluorescence intensity from the upper level [Wade *et al*, 2003]. Experimental data has shown to be in agreement with theoretical analysis of the fluorescence intensity ratio and the sensitivity [Collins *et al*, 1998].

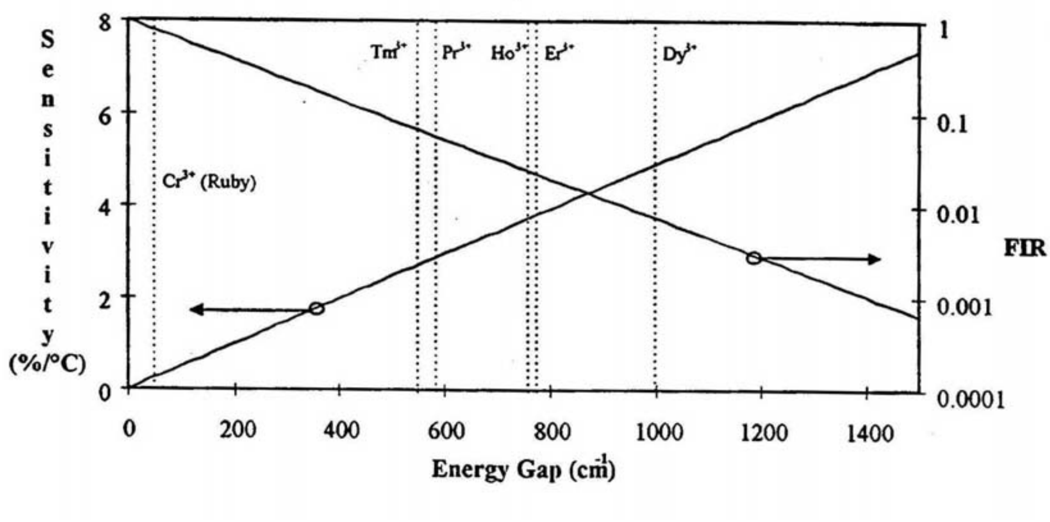


Figure 2.2.2 The change in FIR and sensitivity with respect to the energy gap between the two thermalising levels at room temperature for several rare earth ions [Baxter *et al*, 1996].

2.3 DESIRED CHARACTERISTICS OF A SENSOR FOR THE FLUORESCENCE INTENSITY RATIO TECHNIQUE

The possibility of a commercial sensor based on the fluorescence intensity ratio technique is a real one. A commercial sensor would need to be precise for a wide temperature range, simple to operate, and priced at a reasonable figure. A key area of investigation is of rare earth ions doped in different hosts to determine their viability for use as a sensor [Wade *et al*, 1998; 2003]. The chief feature required of the dopant is that a pair of energy levels is strongly coupled so that their populations follow a Boltzmann distribution so that the fluorescence intensity ratio equation [(1) in Section 2.2] can be applied and render the system independent of source intensity fluctuations. The rare earth ion, the host and the method of pumping the fluorescence, determines the amount of fluorescence intensity that can be obtained from certain energy levels. Generally, a larger energy gap between a pair of thermally coupled levels, achieves a greater variation of the FIR. However, this also results in a reduction in the relative intensity of the fluorescence from the upper level. Practically, better resolution of temperature can be achieved by the appropriate choice of energy levels. One way of minimising price is the use of low-cost commercial laser diodes (or LEDs) and detectors able to respectively excite and detect the fluorescence. Additionally, the choice of the rare earth ion and the host substance impacts on all of these requirements. The host substance normally controls the maximum operating temperature and indeed the effective temperature range of the sensor. Different hosts (for example, silicate glasses) may broaden fluorescence bands (as compared to crystal hosts), with only a minor effect on the absorption and fluorescence wavelengths. Other parameters that need to be considered are the doping concentration of the fibre, and the core size of the fibre. These are discussed in Section 3.3.

Table 2.3.1 summarises the desired features of the sensor from studies into the rare earth ions and different hosts. Where it is appropriate, specific quantitative requirements for silica fibre hosts are detailed since a neodymium doped silica fibre sensor was used for the window and fire experiments.

Table 2.3.1 Desired characteristics of rare earth dopants and host materials (and quantitative requirements for silica fibres) for use in fluorescence intensity ratio sensing systems as reported by Wade *et al* [1998; 2003].

Desired Characteristic	Requirements of Sensing Material
Maximised temperature resolution.	Large energy gap between thermalising levels so as to achieve a greater variation of the ratio over a certain temperature range. Mostly dopant ion dependent.
For a pair of energy levels, the population of the upper level is sufficient so as to discriminate from noise in the sensor (particularly at low temperatures).	Energy levels must possess an energy gap of $< 2000\ cm^{-1}$ to ensure a sufficient transition rate. Determined by dopant ion and host.
Distinguishable fluorescence peaks of separate wavelengths.	The pair of energy levels are to be separated by $> 200\ cm^{-1}$ so that fluorescence wavelengths are not significantly overlapping. Influenced by the pair of excited levels and the host substance.
Transitions must be largely radiative so that sufficient fluorescence intensity from an energy level can be obtained.	The pair of thermally linked energy levels are $\geq 3000\ cm^{-1}$ above the next lower energy level, to minimise non-radiative transition rates.
Fluorescence able to be measured with widely available detectors.	Energies of levels are between 6000 and 25000 cm^{-1}.

2.4 OVERVIEW OF THE DEVELOPMENT OF THE FLUORESCENCE INTENSITY RATIO TECHNIQUE

It was Berthou and Jorgensen [1990] who initially considered the employment of the fluorescence intensity ratio technique with a rare earth doped fibre temperature sensor. The system used erbium and ytterbium co-doped into a heavy metal fluoride glass as the sensing material. The excitation source was of a wavelength close to 980 nm. However, the realisation of this system to a commercially feasible temperature sensor was considered improbable, as 980 nm laser diodes were expensive at the time. A further drawback was the relatively narrow useful temperature range of the system. At temperatures above 150 °C, there was a drastic drop in fluorescence intensity from the excited state levels, and the glass transition temperature is low.

Baxter *et al* [1996] sought to develop this work by building a prototype sensor with a commercially viable pumping scheme. Table 2.4.1 lists the various rare earths doped in either silica or ZBLA host materials (with praseodymium doped in both hosts for comparison) that were considered for their suitability in a FIR based temperature sensor. In the case where the fluorescence intensity was inadequate from one or both levels, work on these materials was halted. Praseodymium (excited with a 467 nm single line argon ion laser) and erbium (pumped at near 800 nm) were studied further. In addition, erbium co-doped with ytterbium in silica fibre using a 980 nm pump source was investigated. The use of a silica fibre host (as opposed to a ZBLA glass host [Berthou and Jorgensen, 1990]) allowed for a higher temperature range (up to 600 ºC) to be used. These samples were considered to be suitable in a FIR based temperature sensor. Comprehensive details of this investigation can be found in the reference.

Table 2.4.1 Rare earth materials and the designation of the energy levels and the appropriate energy difference [Baxter *et al*, 1996].

Rare earth	Energy levels investigated	Energy difference (cm^{-1})	Host material
Praseodymium	$^3P_0$ & $^3P_1$	~580	ZBLA, silica
Dysprosium	$^4F_{9/2}$ & $^4I_{15/2}$	~1000	Silica
Holmium	$^5F_3$ & $^5F_2$	~760	ZBLA
Erbium	$^4S_{3/2}$ & $^2H_{11/2}$	~780	Silica
Thulium	$^3F_2$ & $^3F_3$	~550	ZBLA

Prior to the work of Baxter *et al* [1996], Maurice *et al* [1995] reported an experimental point temperature sensor using erbium (Er^{3+}) doped silica fibre pumped at 800 nm, as shown in Figure 2.4.1.

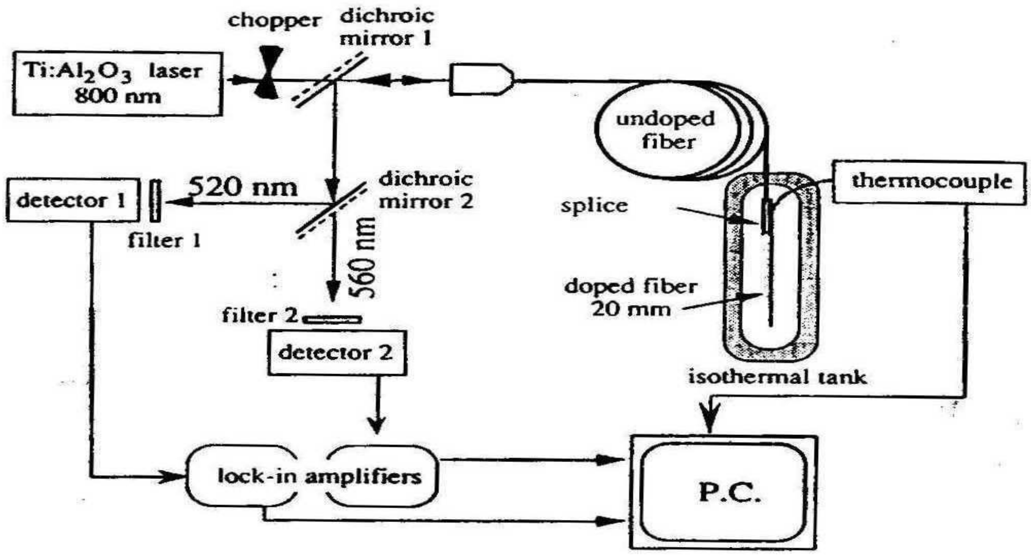


Figure 2.4.1 Schematic of the FIR experimental sensor arrangement of Maurice *et al* [1995].

The green fluorescence intensity of the erbium transitions of $^2H_{11/2}$ and $^4S_{3/2}$ energy levels to the ground state was studied as a function of temperature. Ytterbium-erbium co-doped fibres, pumped at 980 nm, were also included in the study. Available temperature stabilised laser diode sources at wavelengths near 800 nm and 980 nm are suitable for use as practical pump sources in a sensor. The FIR of the sensor was measured at room temperature through to 640 ºC. The standard deviation between the experimental values and fitted data was determined to be 1.1 °C. An investigation into the dependence of fibre length on FIR was also carried out. As was expected, increasing the fibre length produces a non-linear increase in total fluorescence intensity. The self-absorption mechanism that restricts the length of the sensing fibre is described in detail in the reference. In brief, a longer fibre is more vulnerable to excitation source alterations in pump power and wavelength due to these self-absorption characteristics of the doped fibre at green wavelengths. A compromise between greater fluorescence intensity levels and the self-absorption of the doped fibre is required to achieve a functional sensor.

In another body of work, a study of the thermalisation of the $^3P_0$ and ($^3P_1$ + $^1I_6$) energy levels of praseodymium doped ZBLAN glass led to the development of a prototype point temperature sensor [Maurice *et al*, 1997a]. The simplified energy levels of Pr^{3+} in ZBLAN utilised in the sensor are shown in Figure 2.4.2(a). The FIR was measured at temperatures of −60 to 225 °C, and the results are illustrated in Figure 2.4.2(b).

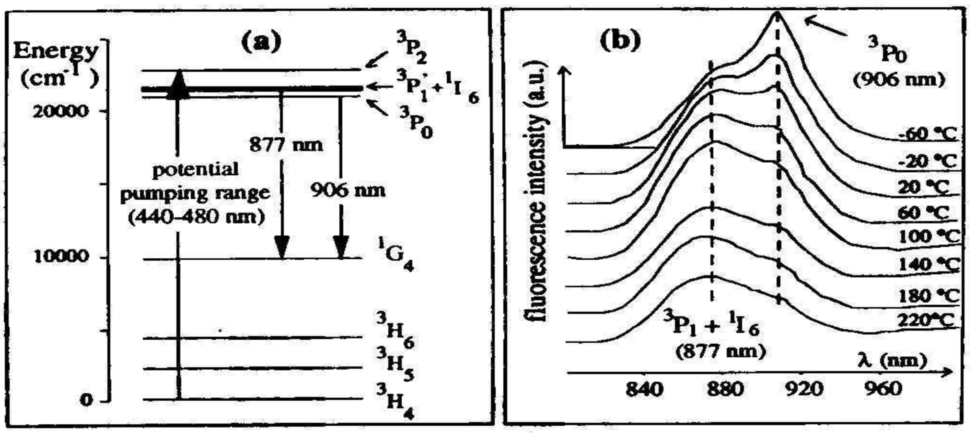


Figure 2.4.2 (a) Simplified energy scheme of Pr^{3+} in ZBLAN (b) Emission of the $^3P_0$ and ($^3P_1$ + $^1I_6$) levels at the shown temperatures [Maurice *et al*, 1997a].

From this work, a prototype sensor was built using a bulk sample of Pr^{3+} doped ZBLAN with a doping concentration of 1%. The sensor was excited by a 2 mW (pump source) blue light emitting diode. Temperature tests were carried out for a temperature range of –45 to 225 °C. The sensor achieved an accuracy of better than 1 °C for this range. This work demonstrated that an inexpensive pump source could be used for a point temperature sensor based on the FIR method.

The FIR method is inherently a self-referencing method, as it does not rely on absolute values of fluorescence intensity. The ensuing advantage is that it is not restricted by fluctuations in the laser pump source. This variance in the excitation source can be seen as a difficulty for other sensing techniques [Voger *et al*, 1993; Wade and Collins, 1994]. Maurice *et al* [1997] presented the advantages of the self-referencing nature of the FIR technique. The self-referencing nature of the sensing device was characterised by measuring the fluctuations of the FIR at room temperature while pump power and pump wavelength were varied, as well as causing bending loss in the fibre. Since it is possible for these features to vary continually in real schemes, the performance of the sensor was characterised when subjected to these changes.

Figure 2.4.3 illustrates the temperature sensor studied by Maurice *et al* [1997]. Ytterbium (Yb^{3+}) doped fibre with a doping concentration of 2000 ppm was used. The thermalising sub-levels utilised were ${}^2F^a_{5/2}$ and ${}^2F^b_{5/2}$ Stark spit levels of ytterbium doped silica fibre. The high fluorescence efficiency of Yb^{3+} allowed the use of an 810 nm AlGaAs laser diode pump source. Based on a fourth order polynomial fit, the sensor demonstrated an accuracy of about 1 °C, and an average deviation of 0.6 °C in the temperature range of 0 to 600 °C. The use of uncoated fibres limited the maximum temperature able to be studied; however the use of an appropriate fibre coating may extend the temperature range below zero and in excess of 600 °C.

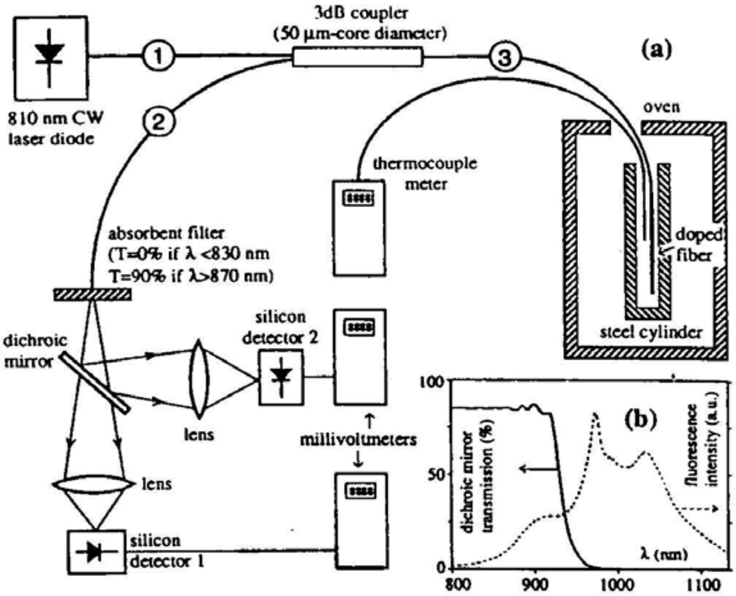


Figure 2.4.3 Ytterbium doped fibre temperature sensor reported by Maurice *et al* [1997].

Studies of prolonged high temperature treatment of the sensing fibre

In regards to fluorescence lifetime temperature sensing studies, the effects of treating the sensing fibre to very high temperatures for a prolonged period of time were mentioned in Chapter 1.2.2. Annealing the doped fibre stabilised the fluorescence lifetime decay dependence, and improved the repeatability of the sensor [Zhang *et al*, 1998; 1998a]. A similar characteristic result was recently reported for the annealing of neodymium doped fibres used in fluorescence intensity ratio sensing [Sidiroglou *et al*, 2003]. For ytterbium doped fibre and common communications fibre, annealing has been shown to produce distinct alterations in the refractive index of each of these fibres [Wade *et al*, 2001b]. The precise reason for these changes is not fully understood [Zhang *et al*, 1998].

In the work of Sidiroglou *et al*, a neodymium doped fibre sample (of length 5 cm) underwent a series of temperature cycling tests, both before the fibre was annealed and afterwards. During these cycling tests and during the annealing period, the fluorescence spectra with respect to temperature were measured to calculate the fluorescence intensity ratio. Firstly, the fibre was repeatedly heated from about 18 ºC to 700 ºC and then cooled to its initial temperature. Next, the sensing fibre was annealed at a temperature of 750 ºC for 100 hours. The initial round of temperature cycling tests was then repeated on the annealed fibre. A final cycling test up to 900 ºC was carried out on the fibre for qualitative comparison with work done by Zhang *et al* [1998]. The effects of annealing have been shown to lead to better reliability of FIR systems: "Annealing the fibre as described has improved the accuracy of the fluorescence intensity ratio verses temperature characteristics from approximately 2.5 ºC to approximately 1.3 ºC."

[Sidiroglou *et al*, 2003]. The Differential Imaging Contrast imaging technique [Dragomir *et al*, 2000] was used to measure the refractive index profiles of the sensing fibre, before and after it had undergone annealing (shown by Figure 2.4.5).

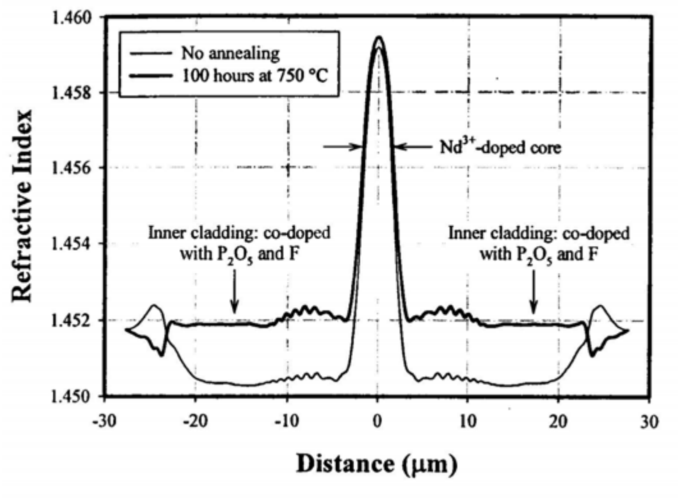


Figure 2.4.5 Refractive index profiles of the core of neodymium doped fibre, before and after annealing as detailed by Sidiroglou *et al* [2003].

2.5 STRAIN EFFECTS IN FLUORESCENCE INTENSITY RATIO TEMPERATURE SENSING

In general most devices used in the measurement of strain or temperature are also sensitive to changes in the other parameter. Accordingly, of interest for this work, a particular compensation configuration may be required to ensure that the effects due to strain are not erroneously measured as changes in temperature. An extra compensation arrangement may be needed. Additional complexities to the temperature sensor invariably involve a rise in its cost. Thus, in design and monetary terms, it is highly desirable to have a fibre temperature sensor, which is independent of strain effects. The fluorescence intensity ratio technique has demonstrated superlative insensitivity to strain, practically zero [Wade *et al*, 2000], making it a most attractive temperature measuring choice for use in settings that are susceptible to strain. Rather than preclude the technique from use in a dual temperature and strain sensor, this non-sensitivity to strain has meant that the fluorescence ratio technique may be used as a temperature reference in a dual temperature and strain fibre sensor [Collins *et al*, 2002].

Investigations into the influence of strain on the measurement of temperature have been performed using ytterbium, neodymium- and erbium doped fibres [Wade *et al*, 2000a; 2001; Trpkovski *et al*, 2003]. For these fibres, a temperature error of 0.4 ºC or less was measured for a strain application of 1000 $\mu\varepsilon$. (For erbium doped fibre of 1000 ppm and 6 cm in length, a temperature error of 0.04 ºC was reported [Trpkovski, 2003].) In practical terms, the near-zero strain sensitivity can be considered negligible with respect to the noise of the sensor. A difficulty with other existing temperature measuring systems is their cross sensitivity to strain [Jones, 1997]. The temperature errors of the FIR systems are less than those reported for other sensors (for an applied strain of 1000 με). For example: fluorescence lifetime (temperature errors of 1.6 ºC to 6 ºC) [Sun *et al*, 1998a; Collins *et al*, 2002]; Brillouin intensity method (3 ºC) [Lees, 1999], Polarimetric (7 ºC) and Fabry-Perot (87 ºC) [Kersey *et al*, 1997]; and fibre Bragg gratings (100 ºC) [Rao, 1997]. As such, the fluorescence intensity ratio method is ideal for the temperature tests of window glass performed in this body of work. Temperature measuring techniques that are cross sensitive with strain are unsuitable because of the occurrence of significant thermal expansion of window glass during the fire simulations.

The strain and temperature investigation by Trpkovski *et al* [2003] provides the basis for the realisation of a dual sensor utilising the FIR technique. The dual sensor is a fibre Bragg grating spliced to erbium doped fibre (about 12 cm in total length). The sensor is most suited for point sensing applications where restrictions on space may be an issue. Its use in further fire research could be possible should multiplexing of the sensor be developed.

2.6 APPLICATION

A particular industrial application that demonstrates the advantage optical sensing can have over thermocouples was described by Wade *et al* [1996]. The FIR based sensor used ytterbium doped fibre to measure the temperature of optical fibres in optical power ground wire (OPGW) cables, manufactured by the former Olex Cables (Tottenham, Victoria). The temperature measurements were made during fault current testing to assess the OPGW's performance and reliability. The temperature of the metallic outer covering of the OPGW can be measured using thermocouples. However, measuring the temperature near the optical fibres of the OPGW cable has been more difficult. Thus, a 5 cm length of ytterbium doped sensing fibre (doping concentration of 350 ppm in an 11 μm core) was fusion spliced into standard communications fibre, which was integrated into the OGPW at the manufacturing stage. Figure 2.6.1 shows the experimental arrangement used to measure the thermal response of the OPGW cable.

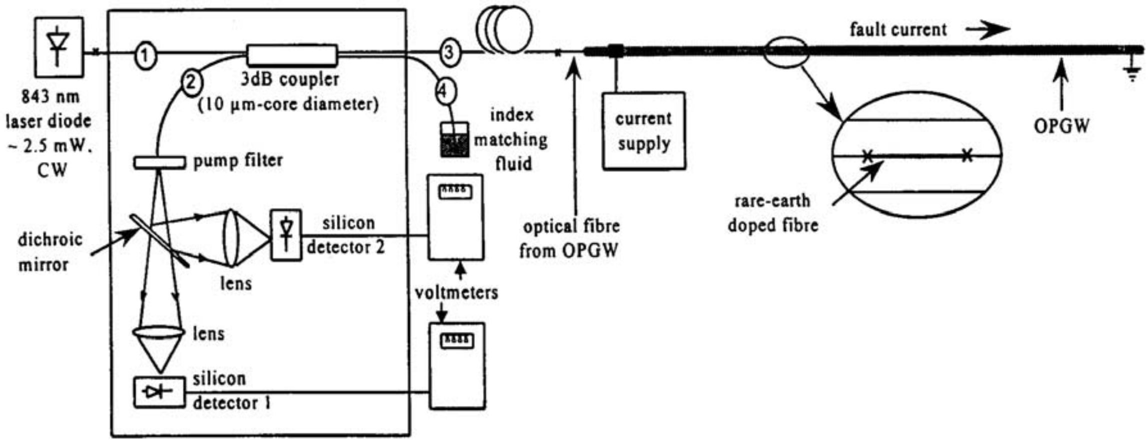


Figure 2.6.1 Experimental arrangement used to measure the thermal response of OPGW cables [Wade *et al*, 1996].

A fault current simulation usually exposes the OPGW to a current pulse of 20 kA rms for 0.45 seconds. Figure 2.6.2 shows a typical temperature result for a fault current test of the OPGW cable, and compares it with the temperature measured by the FIR sensor. It was reported that the noise was mainly from the electronics of the sensor's detectors.

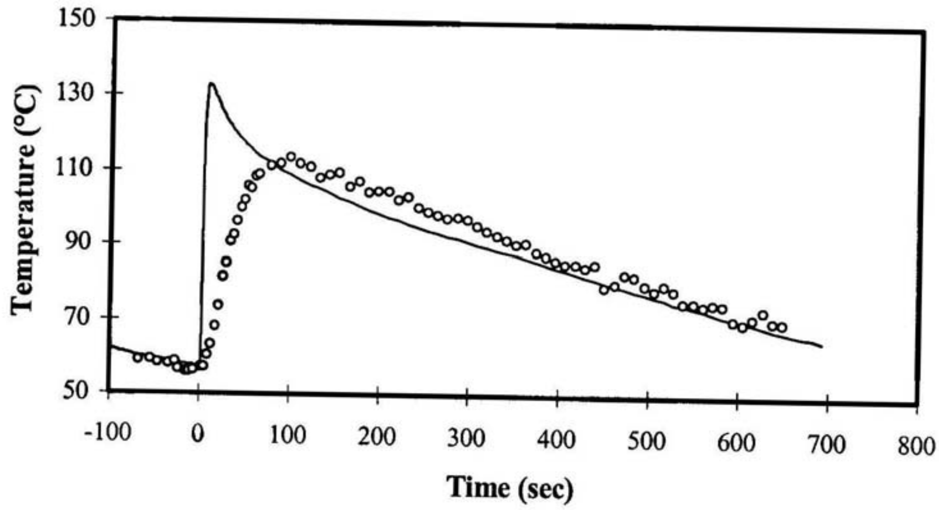


Figure 2.6.2 Optical fibre temperature (depicted by the circles) and OPGW cable temperature (solid line) during OPGW fault current test [Wade *et al*, 2003].

2.7 CONCLUSION

The fluorescence intensity ratio technique has been shown to be an effective and valid optical system for measuring temperature. Although still under development, particularly with respect to dual temperature and strain applications, the FIR technique has been established as a temperature sensing scheme that is supported by its advantages over other optical techniques, such as short length, fast response time, negligible strain sensitivity, broad operating temperature range, and simple design configuration. These advantages, along with the scope to adhere bare rare earth doped fibre directly to the surface of window glass, justifies its choice for the investigations conducted in this thesis.

Chapter 3

Characteristics of Neodymium for the Fluorescence Intensity Ratio Technique

3.1 INTRODUCTION

This chapter endeavours to give the reasons for the appeal and use of neodymium doped silica fibre for the fluorescence intensity ratio technique. Neodymium doped in silica fibre has been demonstrated as one of the most suitable for temperature sensing. A description of these fibres is included here as they were used as the sensing probe for the research of window glass breakage pertaining to this thesis.

3.2 GENERAL CHARACTERISTICS OF NEODYMIUM

It is well known that it is the electronic configuration of the rare earths, which so qualifies them as important fibre dopants for use in temperature sensing, and their original area of use in lasers and amplifiers [Riseberg and Weber, 1976; Miniscalco, 1997]. The suitability of a material for thermal sensing is dependent upon the traits of both the host substance and the dopant ion. Comparative studies of a number of rare earth doped materials for the fluorescence intensity ratio method has been reported [Wade *et al*, 1998; 2003]

In neodymium doped materials, a pair of thermally coupled energy levels from which fluorescence is induced are the $^4F_{3/2}$ and $^4F_{5/2}$ levels. Fluorescence terminating at the $^4I_{9/2}$ ground level has been found to be the most appropriate for fluorescence intensity ratio purposes, and was the level utilised in the sensor employed for this research. Wade *et al* [2003] determined that fluorescence terminating at $^4I_{11/2}$ or $^4I_{13/2}$ were more susceptible to overlapping of the emission peaks. The energy separation of the fluorescing $^4F_{3/2}$ and $^4F_{5/2}$ levels is about 1000 cm^{-1}, which is a suitable gap to achieve adequate temperature sensitivity. Also, the separation between the $^4F_{3/2}$ level and the next energy level below is 5500 cm^{-1} ensuring that the decay is predominantly radiative (see Chapter 2.3). Figure 3.2.1 is an energy level diagram of neodymium ions showing the excited state levels and terminating levels presented by Wade *et al* [2003].

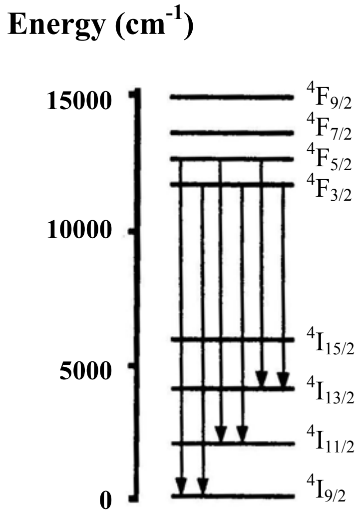


Figure 3.2.1 Energy level diagram of neodymium ions showing the energy levels and pairs of fluorescence transitions from excited state levels as reported by Wade *et al* [2003].

3.3 NEODYMIUM DOPED SILICA FIBRES

Wade *et* al [1998] investigated the fluorescence intensity ratio for a number of neodymium doped host materials. Figure 3.3.1 compares the FIR for these samples.

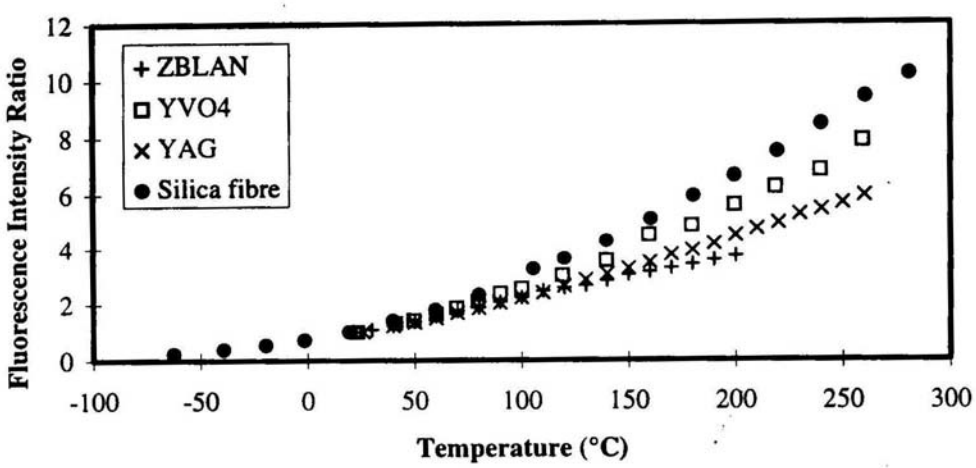


Figure 3.3.1 Normalised fluorescence intensity ratio values with respect to temperature for neodymium in various samples. (FIR values were normalised by taking the quotient of the FIR at each temperature and the FIR at room temperature.) [Wade *et al,* 1998].

One of the most promising samples for use in a FIR based sensor was a bulk sample of neodymium doped yttrium aluminium garnet (YAG). It demonstrated the highest melting temperature of the materials analysed, high fluorescence cross-sections and substantial separations of peaks. It is also readily available. The most favourable sensing material was neodymium ions doped in silica fibre. It demonstrated highest sensitivity of the samples. Other advantages mentioned were its ability to be spliced to regular optical fibre, its effective temperature range, and its widespread availability. The fibres have been studied at a temperature range of typically −50 to 500 ºC with a temperature resolution of 3.4 ºC or less. Another study by Wade *et al*

[1998a] found ytterbium doped silica fibre to also be a promising sensing material because of its similar advantages to neodymium doped fibre.

The investigations by Wade *et al* [1998; 2003] used neodymium doped fibres of different core sizes, doping concentrations and fibre lengths. Naturally, the excitation and detection systems, as well as the application of the sensor may determine the choice of these parameters. The core size must be appropriate so as to allow for efficient coupling of the excitation source as well as be compatible with any standard fibre used in the sensor configuration. The ion doping concentration in the core of the fibre must be sufficient so as to achieve the required amounts of fluorescence, which in particular must able to be distinguished from noise in the detection system. The length of the fibre may be an issue for a particular task. Should a shorter length of fibre be required, generally enough fluorescence intensity can be obtained by appropriately increasing the doping concentration of the fibre, although this may not always be the case.

Figure 3.3.2 shows the fluorescence spectra at different temperatures obtained from the temperature sensor used in the fire research. A 25 cm length of neodymium doped fibre with a core diameter of about 5 μm and a doping concentration of 500 ppm was used to obtain the spectra [Wade *et al*, 1999].

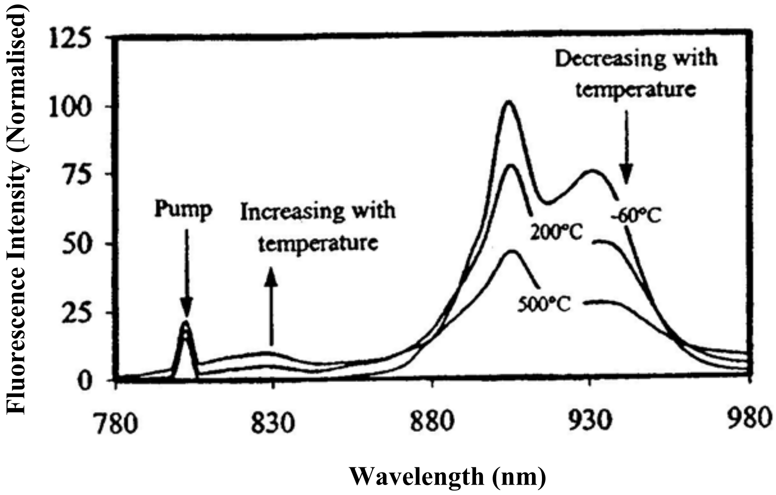


Figure 3.3.2 Fluorescence spectra at different temperatures measured by the temperature sensor used in the fire research. (The spectra were normalised to the peak fluorescence intensity at –60 °C.) [Wade *et al*, 1999].

Chapter 4

Window Glass Temperature Measurement with a Fluorescence Intensity Ratio Fibre Sensor

4.1 INTRODUCTION

This chapter details the experiments in which a fluorescence intensity ratio sensor was applied to the measurement of window glass temperature during fires. A short review of the measurement of window glass in fires was presented in Chapter 1.3. Briefly, the role of window glass in compartment fires is of major significance, since breakage of the windows can mean an escalation of the danger and damage caused. The modelling of window behaviour in fires is limited by the utilisation of thermocouples to measure the window glass temperature. The consequence of dissimilar thermal properties of thermocouples and glass is that compensatory measures must be taken to determine the temperature at which the glass fractures. The application of optical fibres, by means of the fluorescence intensity ratio method, to measure window glass temperature as reported in this thesis, has indeed shown a temperature difference compared with thermocouples. It is suggested that the optical fibre sensor demonstrates a more faithful representation of the window glass temperature.

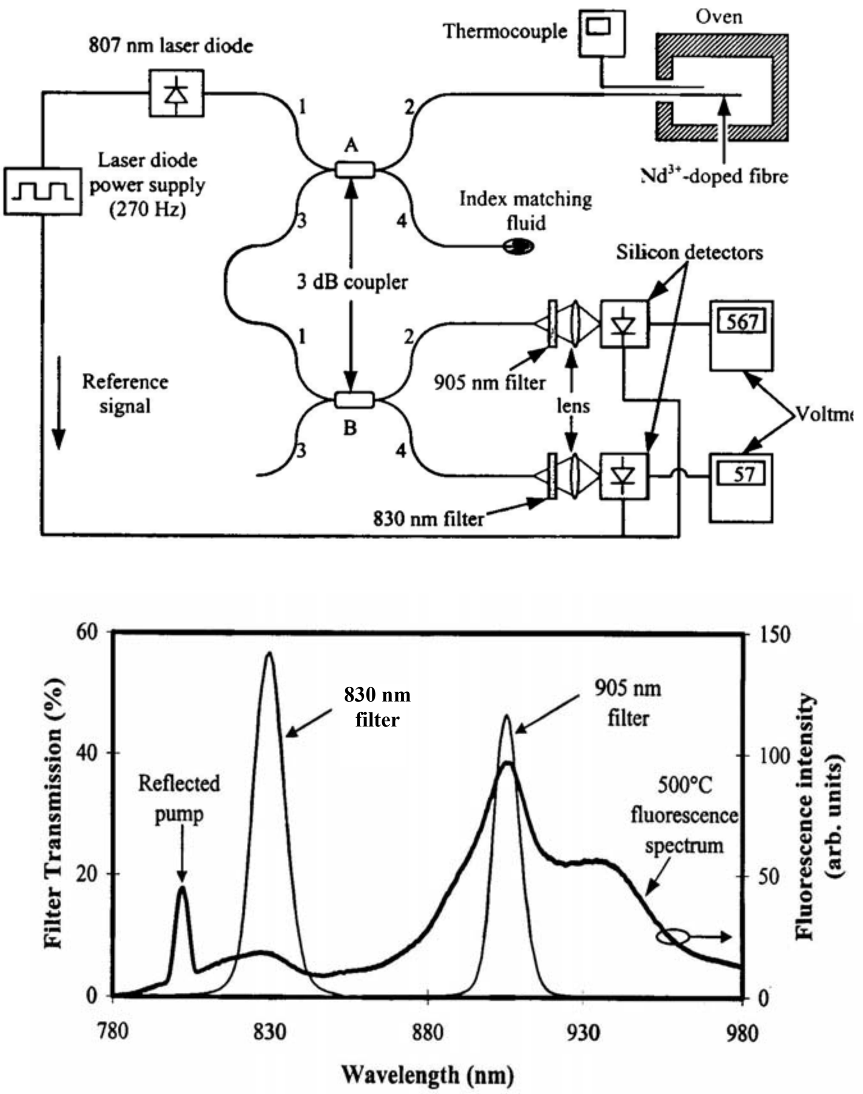
Fire simulations were carried out in a specially built fire chamber to measure the window glass temperature. These fire tests simultaneously catered for two research projects. One being the employment of the fluorescence intensity ratio sensor for window glass measurement. Iraida Khanina of CESARE undertook the other research project, which involved understanding the mechanisms of glass breaking in a fire environment and designing the fire chamber. The fire simulations also produced data to ascertain the relationship between the breakage of windows and non-uniform temperature distribution because of the irregular heating of the windowpane. It also included formulating the associations between the breakage of glass and other factors (hot gas temperature, window dimensions and shading, as examples). Some of the details of this research may be found in Khanina *et al* [2000].

Muscat [1997] and Wade *et al* [1999] reported the development of a prototype neodymium doped optical fibre fluorescence intensity ratio sensor. A version of this prototype sensor was used to measure the temperature of the window glass. Window temperatures measured with thermocouples were also recorded for comparison with the fibre sensor. Bench scale tests to compare the fibre sensor and thermocouples when exposed to radiant heat were undertaken to establish a proof of principle for this research. A number of adhesives to attach the fibre and thermocouples to glass have also been assessed. This small scale work was succeeded by experiments in the specially built fire chamber. A description of the sensor and the course undertaken to enable the use of the sensor for the window glass temperature measurements including details of these measurements and the fire environment follow in this chapter.

4.2 EQUIPMENT DETAILS OF THE FLUORESCENCE INTENSITY RATIO SENSOR PROTOTYPE

4.2.1 A General Description

For the comparative studies of thermocouples and fibres measuring the temperature of window glass, the neodymium doped fibre sensor reported by Wade *et al* [1999] was used. For calibration over the temperature range of –50 to 500 ºC this prototype sensor demonstrated an accuracy of about ±3 ºC. The sensing probe was 59 cm in length, with a doping concentration of 500 ppm and a core of 5 μm diameter. In the window glass breakage work, neodymium doped fibres with different parameters to these were used (as is described in the next section). Figure 4.2.1(a) illustrates the configuration of the sensor. The neodymium doped fibre probe was spliced to port 2 of a 850 nm, 3 dB fibre coupler (depicted by A) and the laser diode excitation source was launched through port 1 via a pigtail. Port 4 is immersed in index matching fluid to eliminate undesirable reflections from the light source. The counter propagating fluorescence through port 3 is separated by means of coupler B. Two bandpass filters allowed fluorescence of two particular wavelength bands to be then focused onto silicon photodiode detectors. Figure 4.2.1(b) illustrates the transmission spectra of the dichroic filters used and the Nd^{3+} fluorescence spectrum at 500 ºC as measured by Wade [1999].



(a)

(b)

Figure 4.2.1 A variation of the prototype sensor used in the investigation of window glass breakage is shown by (a) the neodymium doped fibre sensor based on the fluorescence intensity ratio technique [Wade *et al*, 1999], and (b) Nd^{3+} fluorescence spectrum at 500 ºC and the transmission spectra of the dichroic filters [Wade, 1999].

Optical Power Source and Detectors

Detailed descriptions of the circuit design and calibration of the laser diode driver, power supply and the detectors are reported elsewhere [Muscat, 1997]. Important aspects of the optical power source and the detectors are summarised here. A PIN silicon photodiode was utilised to detect the fluorescence in conjunction with a specially designed detector circuit to modify the signal. A phase locked loop circuit was employed to synchronise the light source and the detectors. Excitation power of about 550 μW was used from a Sharp laser diode (model LM017 MD0) with a centre wavelength of 807 nm [Wade *et al*, 1999]. In order to minimise noise due to ambient light, the light source was modulated to an overall chopped signal of frequency 270 Hz. The filters used were FWHM 10 nm band pass filters centred at wavelengths of 830 nm and 905 nm. The detectors were designed to capture and amplify the fluorescence levels and minimise noise. A phase sensitive detection system using standard integrated circuits was incorporated.

Neodymium Doped Fibre

The sensing probes were neodymium doped fibres. These fibres were fabricated by the Laboratoire de Physique de la Matière Condensée, Universitè de Nice, France. The fibres were manufactured using a modified chemical-vapour deposition process with the solution doping technique [Townsend *et al*, 1987]. For the window glass breakage studies, fibres with a doping concentration of 450 ppm were initially considered. To enable shorter lengths of fibre to be used without compromising fluorescence intensity, fibres with a higher doping concentration were chosen. The doping concentration of the fibres used was 900 ppm, and the core diameter of the fibre was 5 μm.

4.2.2 Preliminary Work

Before the sensor was used for fire tests, preliminary work was done to prepare the sensor for the comparative temperature measurement tests. This involved minimising the light loss through the fibre connections, fusion splicing the neodymium doped fibre to the sensor and choosing the fibre length. Once these were accomplished, temperature calibration of the sensor was undertaken.

Minimising the light loss through the fibre connections

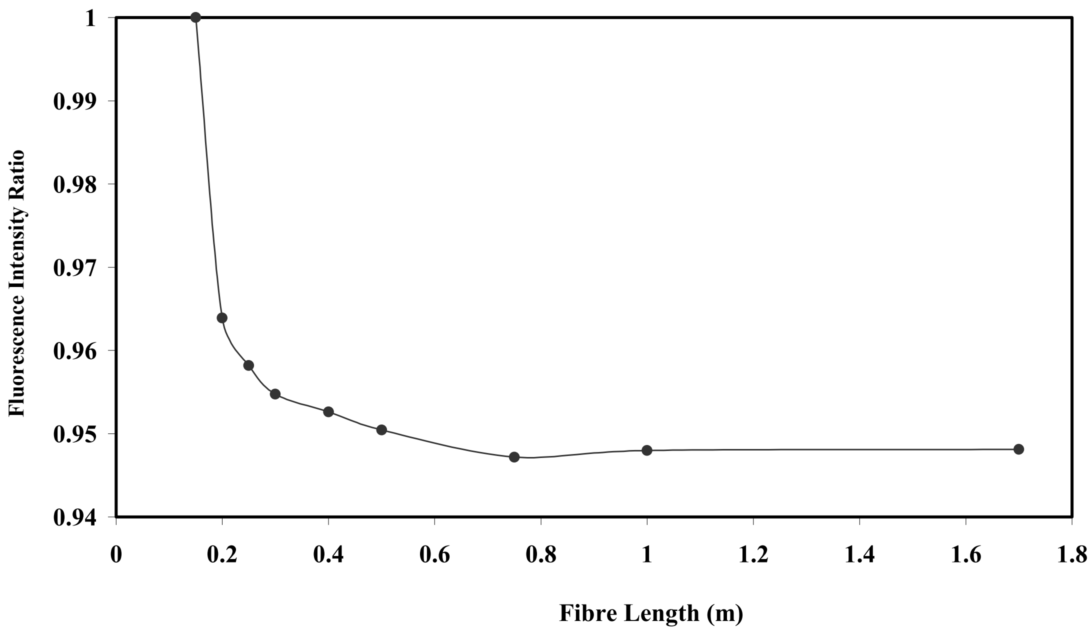
In an attempt to achieve the best possible fluorescence intensity levels, the fibre connections were optimised to minimise splice loss. Referring to Figure 4.2.1(a), a pigtail connection was used for the laser diode to port 1 in coupler A. The free fibre arms of the sensor (port 4 in coupler A and port 3 in coupler B) were index-matched to minimise the reflection back into the laser diode pump source. The wavelength filters were cleaned and fibre arms 2 and 4 of coupler B were cleaved and optimally coupled to the silicon detectors. The neodymium doped fibre was fusion spliced to the sensor (arm 2 of coupler A).

Fusion Splicing of the Neodymium doped Fibre

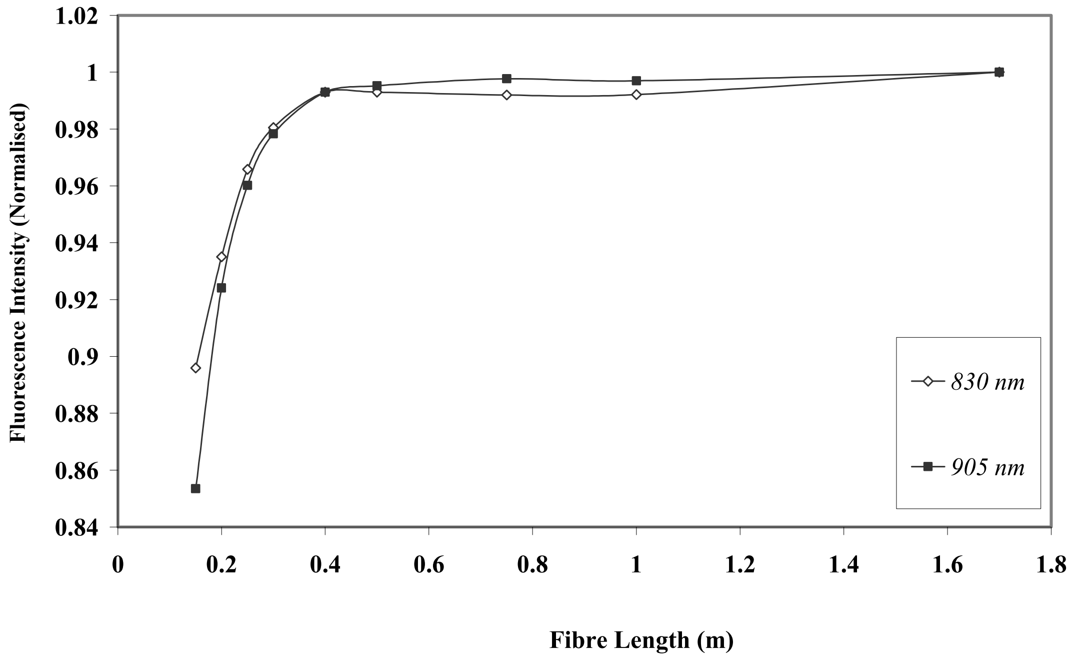
The neodymium doped fibre was fusion spliced to the temperature sensor using a *Siemens* Model A30 fusion splicer. To splice the doped fibre to normal fibre, the parameters used on the fusion splicer were set to those used for standard fibres. The free end of the doped fibre was crushed to eliminate any 'back' reflection of the laser diode pump into the detectors.

Fibre Length Studies

Naturally, a short length of sensing fibre (say a few centimetres) is preferred as it enables a better spatial resolution of the window glass temperature. However, shortening the fibre length will result in a reduction in the fluorescence intensity and therefore signal-to-noise ratio problems may be encountered. Often, the consequence of this is a compromise between fibre length and the spatial resolution of temperature. In the preliminary stages of this work, a *cut-back* of the doped fibre procedure was performed in order to choose a minimal length of fibre, which also enabled sufficient measurement of the fluorescence intensity ratio. The starting length of the doped fibre was just over 1 m. The cut-back procedure also involved crushing the free end of the doped fibre to minimise the back reflection of pump power into the detectors. The fluorescence intensity at the 830 nm and 905 nm wavelengths were measured. The doped fibre was gradually shortened and the above process was repeated. Figure 4.2.2 (a) shows the fluorescence intensity ratio measured for different lengths of fibre and (b) shows the fluorescence intensity at the indicated wavelengths, measured for various lengths of fibre for cut-back studies. The fluorescence intensities were normalised by dividing the fluorescence for each fibre length by the fluorescence at the maximum fibre length. A significant reduction in the FIR was observed for the fibre at 150 mm in length. The reduction in fluorescence intensity at this length indicates that the signal to noise ratio for the detector measuring the 830 nm signal had deteriorated to a point where a further reduction in the fibre length would cause significant problems in obtaining an accurate measurement. To confirm this length, this cut-back procedure was repeated with a new length of fibre (with the same doping concentration of 900 ppm) with consistent outcomes. Thus a length of 150 mm was used.



(a)



(b)

Figure 4.2.2 (a) The fluorescence intensity ratio and (b) the fluorescence intensity at the indicated wavelengths, measured for various lengths of fibre for *cut-back* studies. The fluorescence intensities were normalised by dividing the fluorescence for each fibre length by the fluorescence at the maximum fibre length. (The smoothed lines joining the points are merely to emphasise the changes in FIR with fibre length.)

4.2.3 Temperature Calibration of the Sensor

For each temperature calibration of the sensor, light loss through the fibre connectors was minimised as described in the previous section. The temperature of the laser diode pump source was not controlled. The laboratory in which the calibration of the sensor was done was also not regulated. Ten preliminary calibrations (using the same spliced doped fibre) were performed on the FIR sensor on different days. The calibration curves were incompatible. There are a number of reasons why this might be the case. Some of these seem to be laser diode stability, back reflection to the laser diode, and light from the laser diode entering the detector. These are of concern especially since fluorescence levels at 830 nm are low with respect to system noise [Wade *et al*, 1999]. Another issue was that the laser diode coupling of light into the fibre was subject to drift; this was particularly apparent after a few hours. This drift in the coupling affected the levels of fluorescence intensity possibly due to the excitation of cladding modes.

A comparison of the calibration curves for the fire simulation tests 2, 3, 4, 6 and 8 was made. (The lengths of the doped fibre for these tests were the same). The actual FIR values at 25 °C were considered. The maximum deviation of the FIR value at this temperature was about 50% from the median. The minimum deviation was about 15% from the median FIR value at this temperature. It is interesting to note that the FIR curves for Tests 2 and 3 had the same calibration curves. This variability between the calibrations curves results from the difficulties encountered with the stability of the laser diode, and the coupling of the laser diode and the fibre, when calibrations are performed on different days (and with different doped fibre). Importantly, the slopes of the curves are consistent, and for each separate fire test, a new calibration was performed and the fire tests were performed immediately after calibration of the sensor as described in Section 4.4.

Another possibility for the incompatible calibration curves could be that the doped fibre had not been annealed prior to calibration. At the time that the investigations regarding this thesis were being undertaken, research on the stabilising effects on fluorescence lifetime by subjecting the fibre probe to very high temperatures for an extended time, were in its early stages [Zhang *et al* 1998; and 1998a]. In regards to fluorescence intensity ratio sensing systems, research reported at the time [Wade, 1999] indicated little concern with any annealing effects, especially for the temperature range of interest (from ambient to about 250 °C) in this thesis. Since then annealing the fibre probe has been shown [Sidiroglou *et al*, 2003] to improve the reliability and repeatability of fluorescence intensity ratio sensing systems, as described in Chapter 2.4.

The difficulties mentioned above represent a significant limitation on the practicability of the sensing system used in this work, as discussed in the concluding chapter. In order to limit the occurrence of issues with laser diode stability, a time frame of about two hours was established, during which the temperature calibration of the sensor and the fire simulation test should be performed. The two research groups were coordinated in order to achieve this.

The stability of the laser diode at room temperature was investigated by measuring the FIR for an extended period. The measured FIR was found to have a standard deviation of 2% over an interval of two hours, the same being found for a four hour period. For a twelve hour time frame, the standard deviation of the FIR at room temperature was 5%.

To prepare the doped fibre for calibration, the coating on the doped fibre was removed by soaking in weak sulphuric acid. 'Bare' fibre eliminated any effects that the coating may have, particularly if the coating cracked during the fire tests. Removing the coating enabled the best match between the composition of the sensor probe and the window glass. To facilitate the cooling and heating of the sensor probe, an aluminium case was constructed. The top view is shown in Figure 4.2.3. The doped fibre was inserted into a close fitting cylindrical cavity along the length of the aluminium case. A rod style thermocouple was also inserted snugly into the case (denoted by thermocouple 1). Its tip was located at approximately the middle of the length of fibre. Small holes were drilled for three K-type thermocouples, which were situated as shown in the diagram.

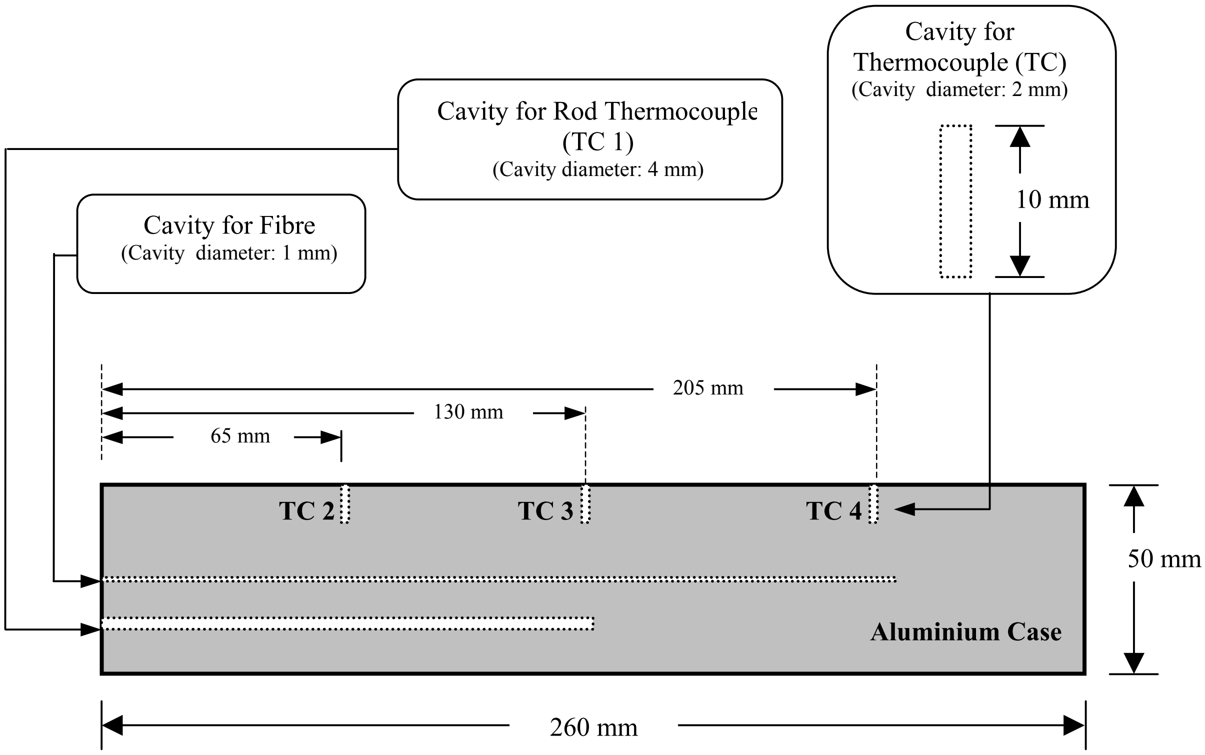


Figure 4.2.3 Cross section of the aluminium case used to heat the neodymium sensor probe and thermocouples, as viewed from the top.

For the temperature calibrations, the encased fibre was placed as shown in Figure 4.2.4. To enable a stable temperature environment to be created for the aluminium case, it was placed in a metal rectangular pipe, which was insulated with plasterboard on three sides. The non-plastered face sat on a thick aluminium block. A household electric hot plate was used to heat the encased fibre at a rate of about 1 °C per minute. The temperature range for the calibration was usually from room temperature to 250 °C, sufficient for measuring the window temperature in a fire environment. The fluorescence levels were measured at the 830 nm and 905 nm wavelengths every 20 seconds. A data logger simultaneously measured the thermocouple temperatures. When the maximum temperature was achieved, the fibre was then cooled to room temperature by gradually lowering the heat from the hot plate. Closer to room temperatures, ice was applied to the aluminium case to assist in the cooling process.

As a side point of interest, the effect of water on the doped fibre was also investigated. Condensation formed when cooling the fibre with ice. A comparison between the fluorescence when the doped fibre was dry and when it was immersed in water was made. The effect of water on the fluorescence was negligible.

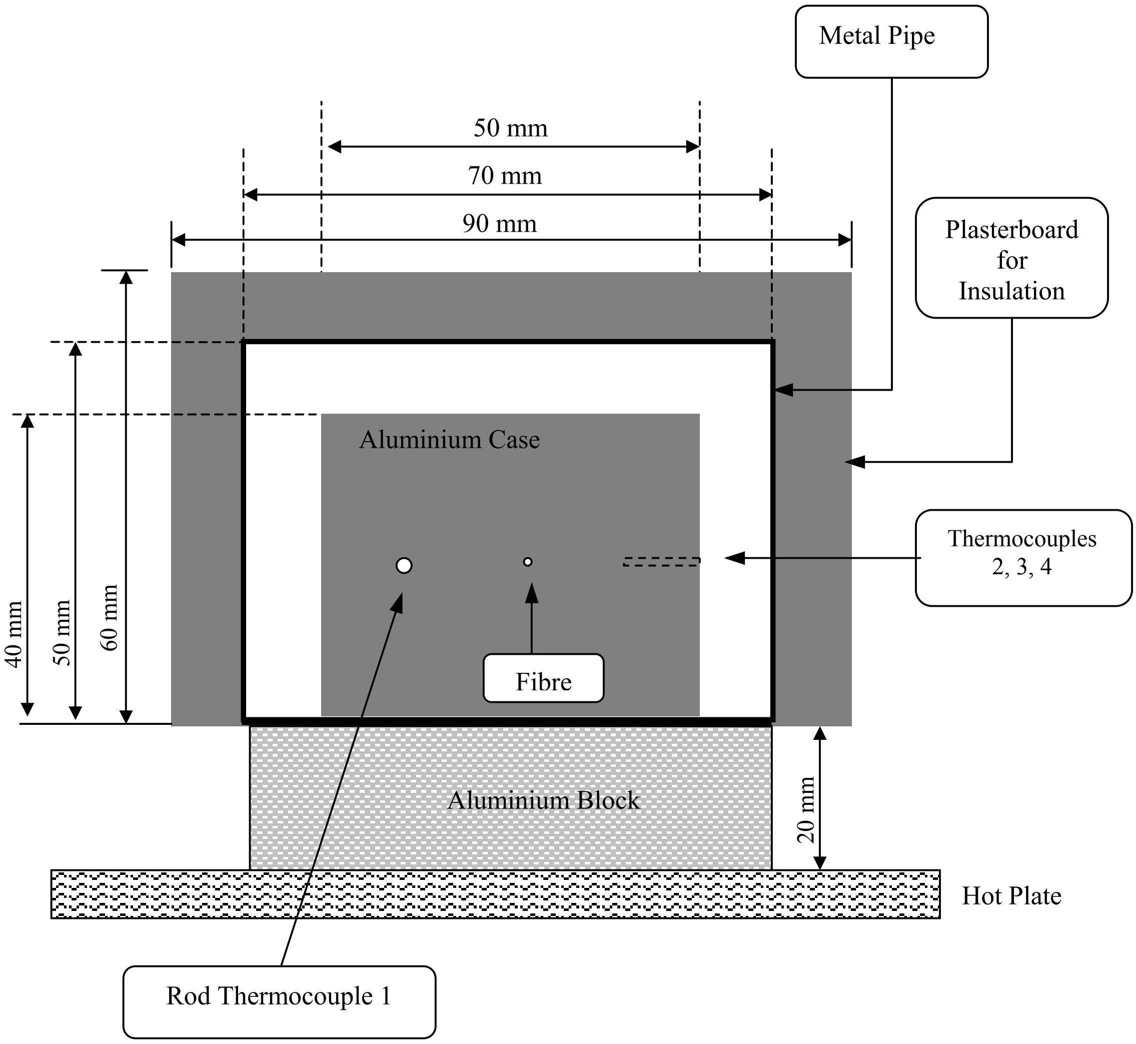


Figure 4.2.4 Front cross-sectional view of the fibre housing on the hot plate for temperature calibration of the fluorescence intensity ratio sensor.

The thermocouples used were commercial K-type chromel alumel thermocouples diameter 1.5 mm with a tolerance of ± 2.5 °C**.** The thermocouples were insulated in Mineral Insulated Metal Sheath (MIMS). This insulation ensured the thermocouples' ability to operate at high temperatures as well as giving protection to the thermocouple from the fire surroundings [Childs, 2001].

Figure 4.2.5 (a) is a typical graph of the temperature of the four thermocouples and their average temperature with respect to time. These temperatures were recorded during a calibration of the sensor with a doped fibre length of 150 mm. Since it is difficult to distinguish each thermocouple temperature in the graph, Figure 4.2.5 (b) shows, as a function of time, the deviation of each thermocouple temperature from the average temperature of the four thermocouples, denoted by $T_{TC} - T_{av}$. The temperatures recorded by the thermocouples differ from the average temperature by less than 2 °C. This average thermocouple temperature can be confidently regarded as representing the temperature of the neodymium doped fibre during the calibration of the FIR sensor.

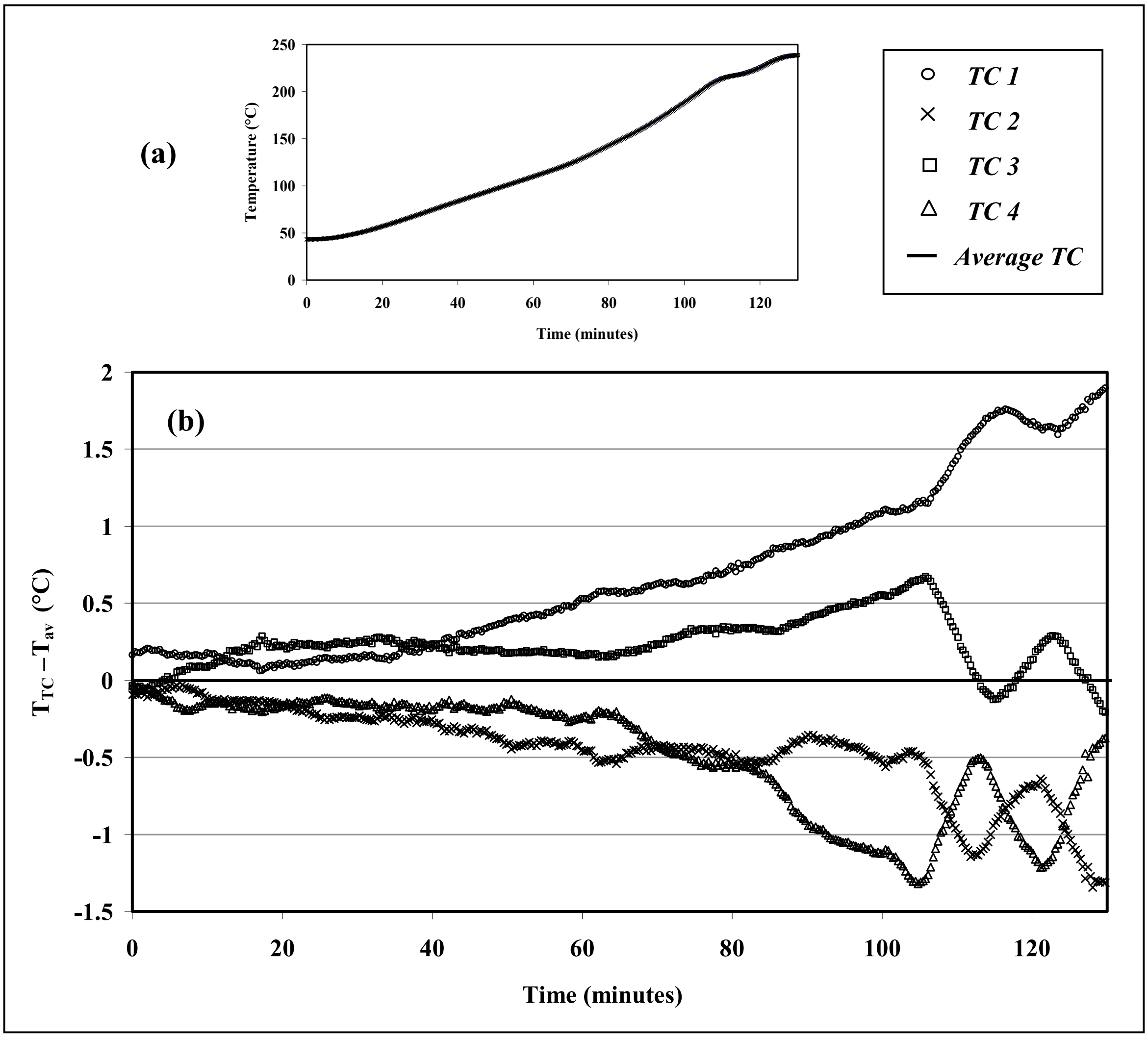


Figure 4.2.5 (a) Typical temperature measurements from the four thermocouples and their average temperature with respect to time and (b) for each thermocouple, the difference between the temperature and the average temperature of the thermocouples, as a function of time. These temperatures were recorded during a calibration of the sensor using the experimental arrangement shown in Figure 4.2.4. (TC = thermocouple.)

Figure 4.2.6 (a) shows a typical calibration result for the neodymium doped temperature sensor, with a doped fibre length of 150 mm. The circles represent the variation of the FIR at the indicated temperatures. The temperature is the average temperature of the four thermocouples used in the fibre holder (refer to Figure 4.2.3). These data were fitted with a fourth order polynomial (solid line), which was then used to create a ‘look up’ or reference table to convert measured FIR to temperature.

Figure 4.2.6 (b) illustrates the temperature error between the average thermocouple temperature and the look-up table values. The look-up table was generated using a fourth order polynomial fit for the temperature range of about 20 to 250 ºC. The standard deviation between the experimentally obtained temperature data and the fitted data was 1.4 ºC, with the maximum error being 5.4 ºC.

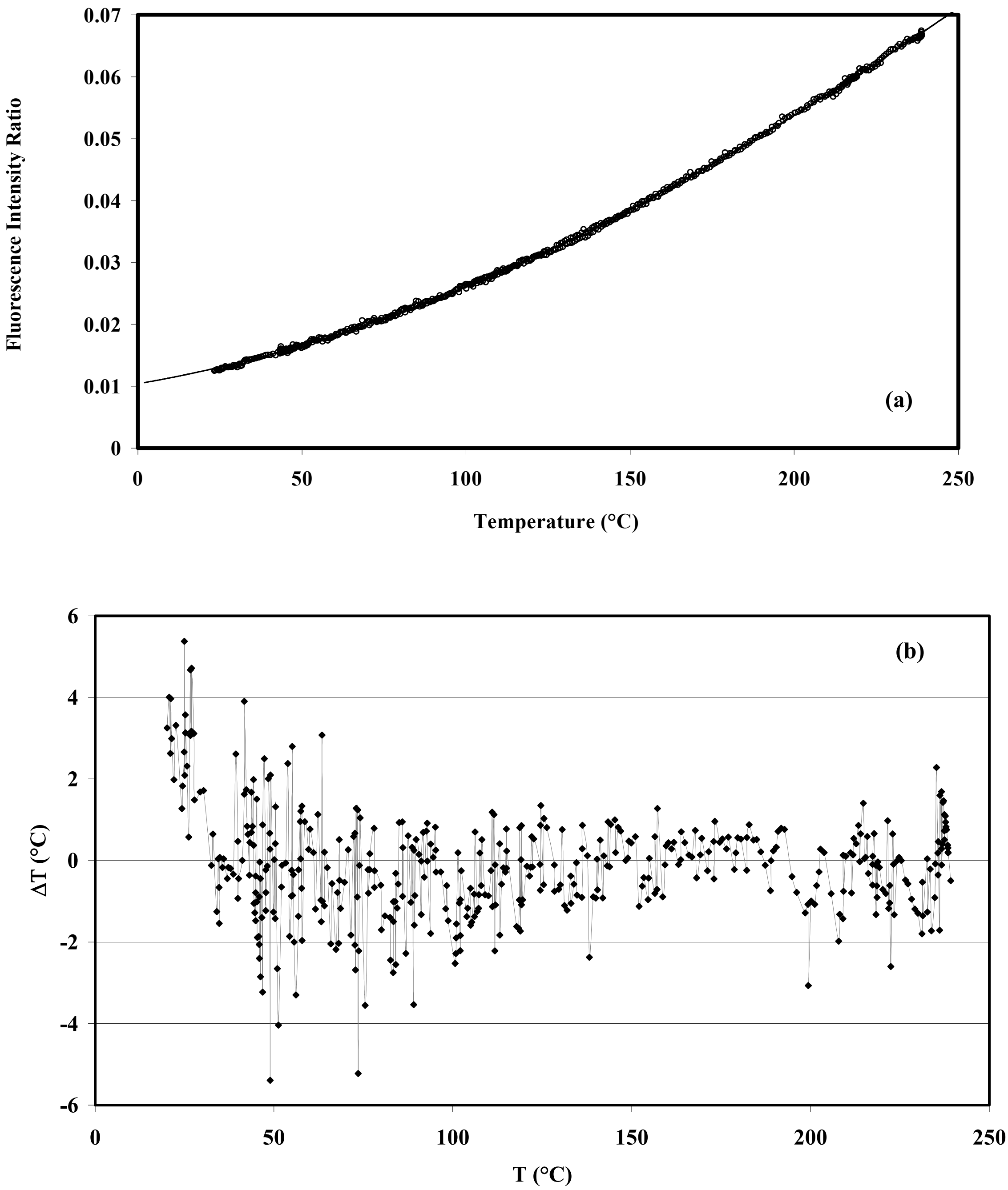


Figure 4.2.6 (a) a typical calibration result of the neodymium doped temperature sensor. (Doped-fibre length is 150 mm). The circles represent the variation of the FIR at the indicated temperatures. This data was fitted with a fourth order polynomial (solid line) and (b) the temperature differences between the experimental data and fourth order polynomial fit. (The lines joining the points are for an aesthetic purpose only.)

4.3 SMALL SCALE RADIANT HEAT TESTS

Temperature tests of the window glass on a small scale were undertaken in order to trial the optical fibre sensor before experiments on a large scale were performed. This stage of work involved assessing various adhesives to attach the fibre to window glass to ascertain their performance in a fire environment. It also included a simulation of fire radiation, comparatively testing the fibre sensor and thermocouples when exposed to radiant heat.

4.3.1 Assessment of Adhesives for the Attachment of Fibre to Glass

Consideration of suitable adhesives to attach the fibre to window glass is important. An ideal adhesive would have no thermal effect on the glass and on the doped fibre. Practically, an adhesive with the minimum impact on the temperature measurement is desired. This is to ensure that it is the temperature of the glass that is being measured, and not the temperature of the adhesive. Other considerations influencing the choice of adhesive were the ease of application of the adhesive, its curing time, its operating temperature, and its appearance when exposed to radiant heat. Standard fibre (about 10 cm in length) was attached to a small piece of window glass using the available adhesives. A Bunsen burner was used to apply a direct flame onto the fibre and glass for about five minutes.

The performance of the adhesives is summarised in Table 4.3.1. These results indicated that *Silastic* silicon rubber, and *KS Bond*, household ‘super’ glue, would be suitable for the fire tests. This is since they successfully attached the fibre to the glass and did not change colour when directly subjected to a flame.

Table 4.3.1 Summary of the performance of adhesives used in the assessment of adhesives for fire tests.

Adhesive	***Initial Colour***	***Rated Operating Temperature***	***Retained Initial Colour***	***Fibre Remained Attached***
Household ‘super’ glue	white	unknown	✓	×
Pyropanel Multiflex Fire-rated acrylic sealing compound	white	unknown	×	✓
Duralco 4462 Epoxy	brown	~250°C	✓	×
Silastic Silicon rubber	clear	<250°C	✓	✓
KS Bond Household ‘super’ glue	opaque	unknown	✓	✓

4.3.2 Exposure of Bare Fibre to Radiant Heat

Thermocouples are usually used to measure the temperature of window glass in fire experiments. The dissimilar material properties of glass and thermocouples mean that they emit, absorb and transmit radiant energy differently. Effectively, thermocouples do not represent the true temperature of window glass in fires.

In order to simulate the heat radiation present in a fire, radiant heat was applied to the neodymium doped fibre sensor probe and thermocouples. It was necessary to ascertain the performance of the fibre sensor with the doped fibre left bare. The next stage was to determine the impact of the glues on the fibre temperature measurements when exposed to radiant heat. For each adhesive a new fibre probe was used. The adhesive was applied along the length of doped fibre and compared with the thermocouples in the radiant heat tests.

Experimental Set Up

Before each radiant heat test was carried out, a 150 mm length of neodymium doped fibre was spliced to the sensor, the light connections were optimised, and the sensor was calibrated in the manner described in section 4.2. A wooden board (with approximate dimensions 120 mm x 300 mm) was used to hold the thermocouples and fibre as illustrated by Figure 4.3.1. Three thermocouples were positioned along the length of the fibre, to enable comparison of the temperature of each of these with the FIR fibre sensor temperature. Small holes were drilled through the board and one thermocouple was inserted through each hole so that the thermocouples protruded just outside of the board. The doped fibre was placed horizontally on the board and held up by three small hooks. The thermocouples and the doped fibre were directly exposed to radiant heat by holding the board in a vertical position. An 1800 W household radiant bar heater (of bar length 40 cm) was then placed about 300 mm in front of the board. The size of the bar heater and its proximity to the thermocouples and fibre were sufficient to ensure that radiative heating was the major heating process. The fluorescence intensity ratio and the thermocouple temperatures were measured every 4 seconds (for about 10 minutes or less). The tests were performed for bare fibre, fibre thinly coated in the *KS Bond* super glue, and fibre coated in *Silastic* silicon rubber.

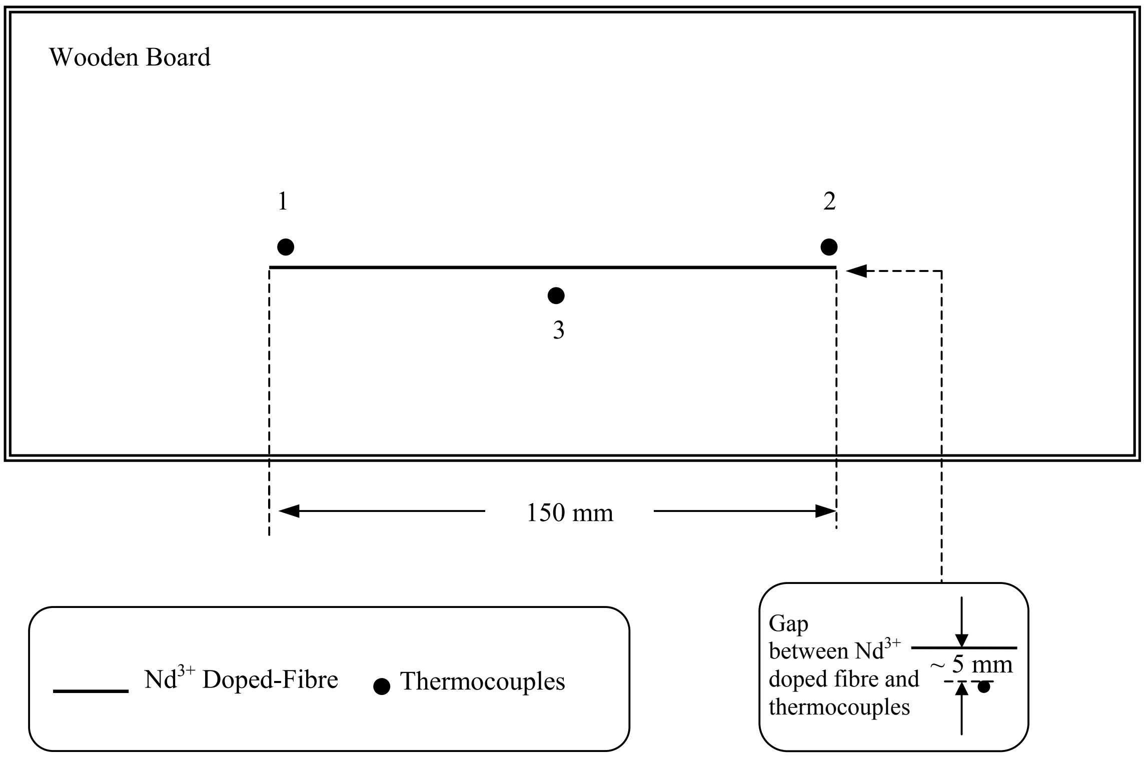


Figure 4.3.1 Set-up of neodymium doped fibre and thermocouples for the small scale radiant heat tests. (Diagram is not to scale.)

4.3.3 Discussion of Small Scale Radiant Heat Tests

Bare Fibre Probe

Figure 4.3.2 is a typical test result of the fluorescence intensity ratio sensor subjected to radiant heat. The doped fibre in this case was left bare. The temperatures of the fluorescence intensity ratio fibre sensor, the three thermocouples and the average temperature of these thermocouples are shown. The recording of temperatures commenced when the household bar heater was switched on. Differences in the temperature of the thermocouples is shown, particularly after 5 minutes, at which stage the bar heater would have been at its maximum operating capacity and the maximum radiant heat would have been applied. The differences in the temperatures of the thermocouples are likely due to uneven heat radiation from the bar heater. The length of the heating element was about 30 cm. As the length of the heater was twice that of the doped fibre, it was thought that the probes were being heated uniformly, given the environment is not as controlled as in a temperature calibration. The heater could have been placed closer for more even radiant heating of the probes, except that this would have produced a more immediate application of radiant heat rather than simulate the increasing radiation of a growing fire in a small room. Although differences in the thermocouple temperatures are apparent, they follow a common trend between them and with respect to the FIR fibre sensor. The FIR sensor indicates a considerably lower temperature than that of the thermocouples. At temperatures above 100 ºC, the doped fibre measures about 25 ºC less than the (average) temperature of the thermocouples. In the application of radiant heat, a significant discrepancy in the measurement of temperature between thermocouples and a bare FIR doped fibre probe has been established.

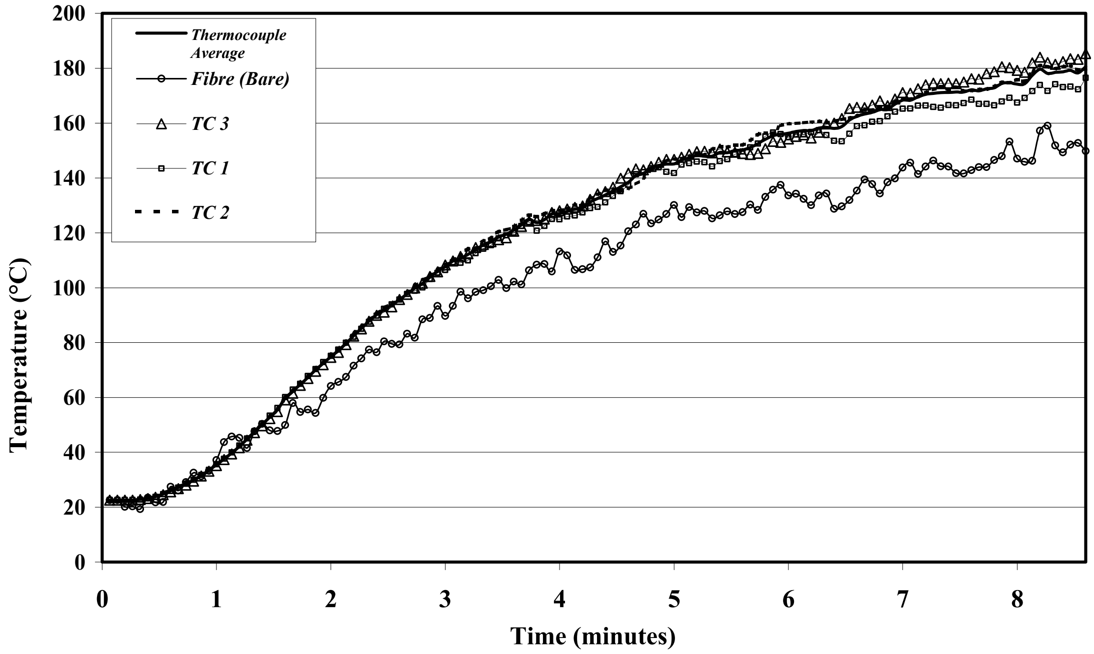
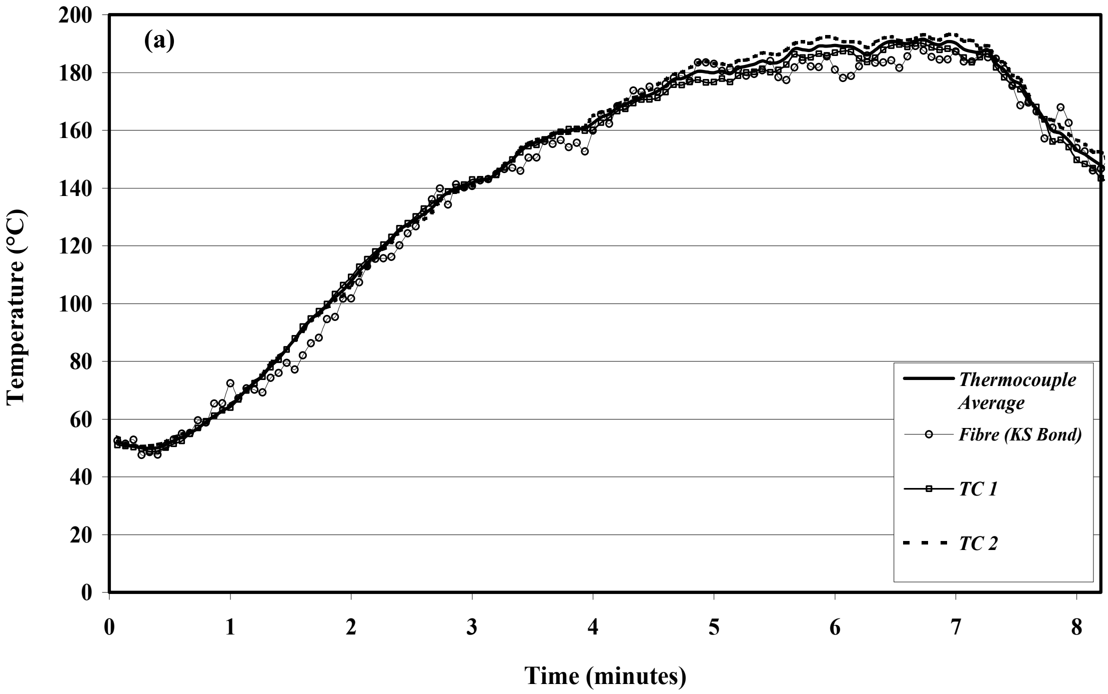


Figure 4.3.2 Comparison of thermocouples and fluorescence intensity ratio sensor exposed to radiant heat in a bench scale test. The neodymium doped fibre probe was bare. (TC = thermocouple.)

Fibre probe coated with an adhesive

Following these small scale radiant heat tests using bare doped fibre, radiant heat tests were undertaken to investigate the performance of the FIR sensor with the doped fibre probe coated in *KS Bond* and *Silastic* adhesives. These were the potential adhesives to be used to attach the doped fibre probe to the window glass for the upcoming compartment fire tests. Figure 4.3.3 illustrates typical results of the covered fibre and thermocouples subjected to radiant heat. The fibre probe was thinly coated with the adhesive and the adhesive was left to cure. The coated fibre was then placed on the board using the same set up as described for Figure 4.3.1. Figure 4.3.3 (a) shows the performance of the neodymium doped fibre probe covered with *KS Bond* super glue with respect to the thermocouples. The results of thermocouple 3 are not shown due to a malfunction in data capture for this thermocouple during these tests. Figure. 4.3.3 (b) shows the temperature as measured by the doped fibre probe covered in *Silastic* silicon rubber compared with the thermocouples. As with the previous radiant heat tests with bare doped fibre, the thermocouples show differences in temperature, but follow similar thermal trends. In regards to the results for the *KS Bond* covered fibre (a), the covered doped fibre shows less of a discrepancy with respect to the thermocouples, than that of the fibre left bare (as seen in Figure 4.3.2). However the temperature trend of the *KS Bond* covered fibre is consistent with the trends followed by the thermocouples. For the *Silastic* coated fibre as shown in (b), the temperature of the fibre is at first generally higher than the temperatures of the thermocouples and then gradually measures temperatures lower than that of the thermocouples. The temperature trend of the fibre probe covered in *Silastic* does not follow the temperature trend of the thermocouples.

Thus the radiant heat simulation tests showed *Silastic* silicon to be unsuitable for use in the fire tests. It was decided that *KS Bond* would be the more suitable adhesive for attaching the doped fibre probe to the window glass to be used in the fire tests. This is due to the consistency of the temperature trends of the *KS Bond* covered fibre and the thermocouples during the radiant heat tests, the validity of which were supported by the confidence given to the accuracy of the calibration of the FIR sensor.



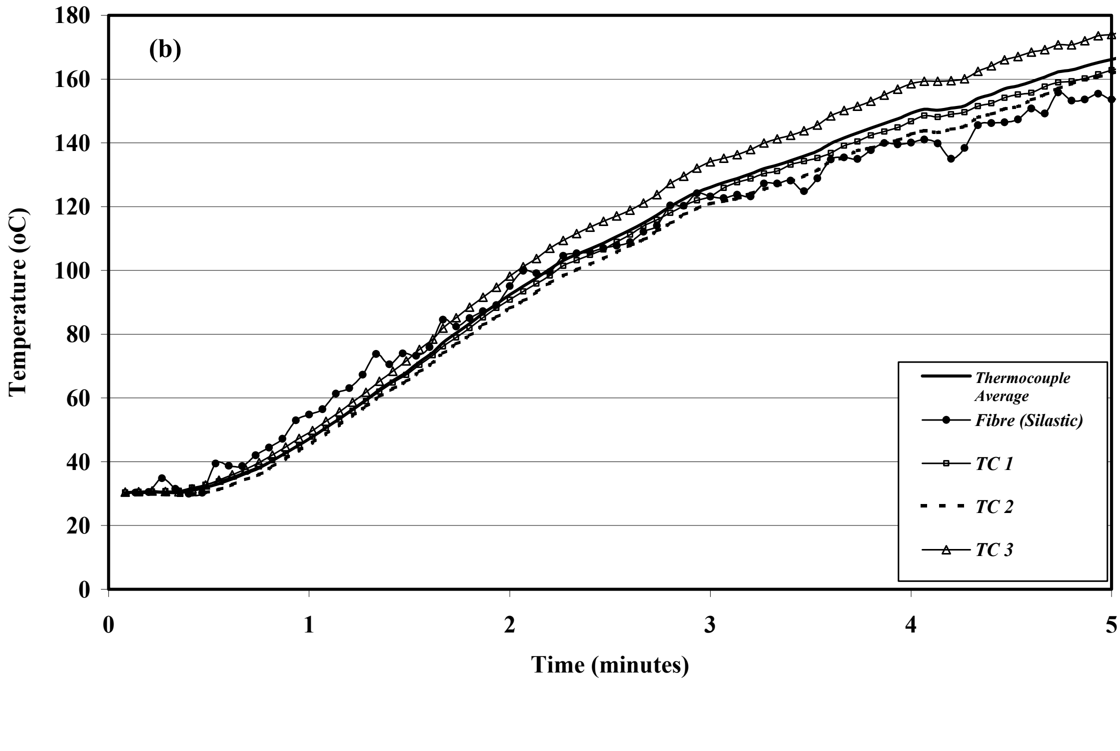


Figure 4.3.3 Performance of fluorescence intensity ratio sensor, with the probe coated in an adhesive. The results are for the fibre probe coated in *KS Bond* super glue shown in (a), and the fibre probe coated in *Silastic* silicon rubber shown in (b). (TC = thermocouple.)

Overall, the small scale tests simulating the heat radiation present in a fire, indicate a clear difference between temperatures measured by bare optical fibre and thermocouples. These radiant heat tests showed the bare doped fibre probe to measure markedly lower temperatures compared to the thermocouples. When the fibre probe was coated with an adhesive such as *KS Bond* and subjected to the same radiant heat conditions, the coated fibre showed the same temperature trends as the thermocouples. For the upcoming fire tests, the advantage that the doped fibre had over the thermocouples would be lost by the application of the adhesive, which was necessary to attach the fibre to the window glass. As is described in the following section, an improved technique to adhere the fibre probe to the windows was introduced for the latter three fire tests. The adhesive was applied at points along the length of fibre, in an attempt to minimise the use of the adhesive and to regain the advantage of the FIR sensor over thermocouples as shown by the small scale radiant heat tests of bare fibre.

4.4 MEASUREMENT OF WINDOW GLASS TEMPERATURE DURING FIRES USING THE FLUORESCENCE INTENSITY RATIO SENSOR

This stage of work involved simulating small room fires and measuring the window glass temperature during the fire. A specially built chamber designed for the work of Iraida Khanina was used. The fire chamber was designed to simulate the start and spread of fire in a small room, and conditioned to ensure that the window glass cracked during the fire. The first series of tests used small windowpanes. Larger windowpanes were used in further experiments. The temperature of the window glass was measured with the fluorescence intensity ratio fibre sensor and thermocouples. An evaluation of the performance of the fibre sensor in a fire environment was made.

4.4.1 Experimental Set Up for the Fire Simulations

The complementary research by I. Khanina regarding the mechanisms pertaining to the breakage of window glass and comprehensive details of the simulation test chamber and the window glass parameters are reported elsewhere [Khanina *et al*, 2000]. In this section, the description of the experimental set up that is included here is intended to give an overall vision of the fire simulation tests with respect to the application of the fluorescence intensity ratio sensor to measure window glass temperature.

Since the fire simulation tests served two areas of research simultaneously, a degree of coordination of the involved workers was required. A schedule was devised to accommodate the necessary calibration of the fibre sensor before a fire test was carried out. The fire test proceeded soon after calibration. Normally, new doped fibre was spliced to the sensor, and the fluorescence intensity ratio sensor was optimised and calibrated as described in section 4.2. The data obtained were quickly checked to ensure that the calibration was satisfactory. The fibre probe was attached to the window glass and left to cure. The window was mounted to the chamber and a fire simulation was carried out. For each completed test, the window was usually damaged and the sensing fibre usually broke on attempts to detach it from the glass, and so a new windowpane was used for each test. The thermocouples were removed from the pane and attached to the new window. A new section of neodymium doped fibre was spliced to the sensor ready for calibration and a new fire test.

The Test Chamber

The inner dimensions of the test chamber were 1.2 m x 1.8 m, with a height of 1.2 m. It included a small opening for ventilation, and a detachable wall to allow for the mounting of one windowpane. Methylated spirits was used as the fuel source and placed in trays on the chamber floor. The trays were ignited in an order so as to control the heat release rate. Logging of the temperature data began when all the trays were set alight, and halted when the fuel was exhausted. Thermocouple and fibre sensor temperatures were recorded every 15 seconds. The gas temperatures of the upper half of the chamber ranged from about 250 to 370 ºC for the major period of the fire [Khanina *et al*, 2000].

The Window Glass

Three fire simulations were performed with small windowpanes. The dimensions of these were 200 mm x 200 mm with a thickness of 3 mm (see Tests 1 to 3 in Table

4.4.1). Five fire simulations followed using larger windows that were 600 x 600 mm in size. Thermocouples were positioned near the fibre. The fibre was attached to the centre of the window glass in a horizontal position. These thermocouples and the fibre sensor were placed on the inside surface of the windows, thus being directly exposed to the fire in the chamber. Figure 4.4.1 (a) shows the placement of thermocouples and doped fibre (length 150 mm) on the small windowpanes as used in the fire simulation tests 2 and 3. Figure 4.4.1 (b) shows the position of the same length of doped fibre for the larger windowpanes as used in fire simulation tests 4 and 6. Similar configurations were used for different lengths of doped fibre used in other fire simulations. A frame (of depth 10 mm) enabled the mounting of the window to the wall and additionally shaded the edges of the windows. As required by the complementary research work, additional shading was used along the edges for some of the windows. Details of the aforementioned parameters are summarised in Table 4.4.1.

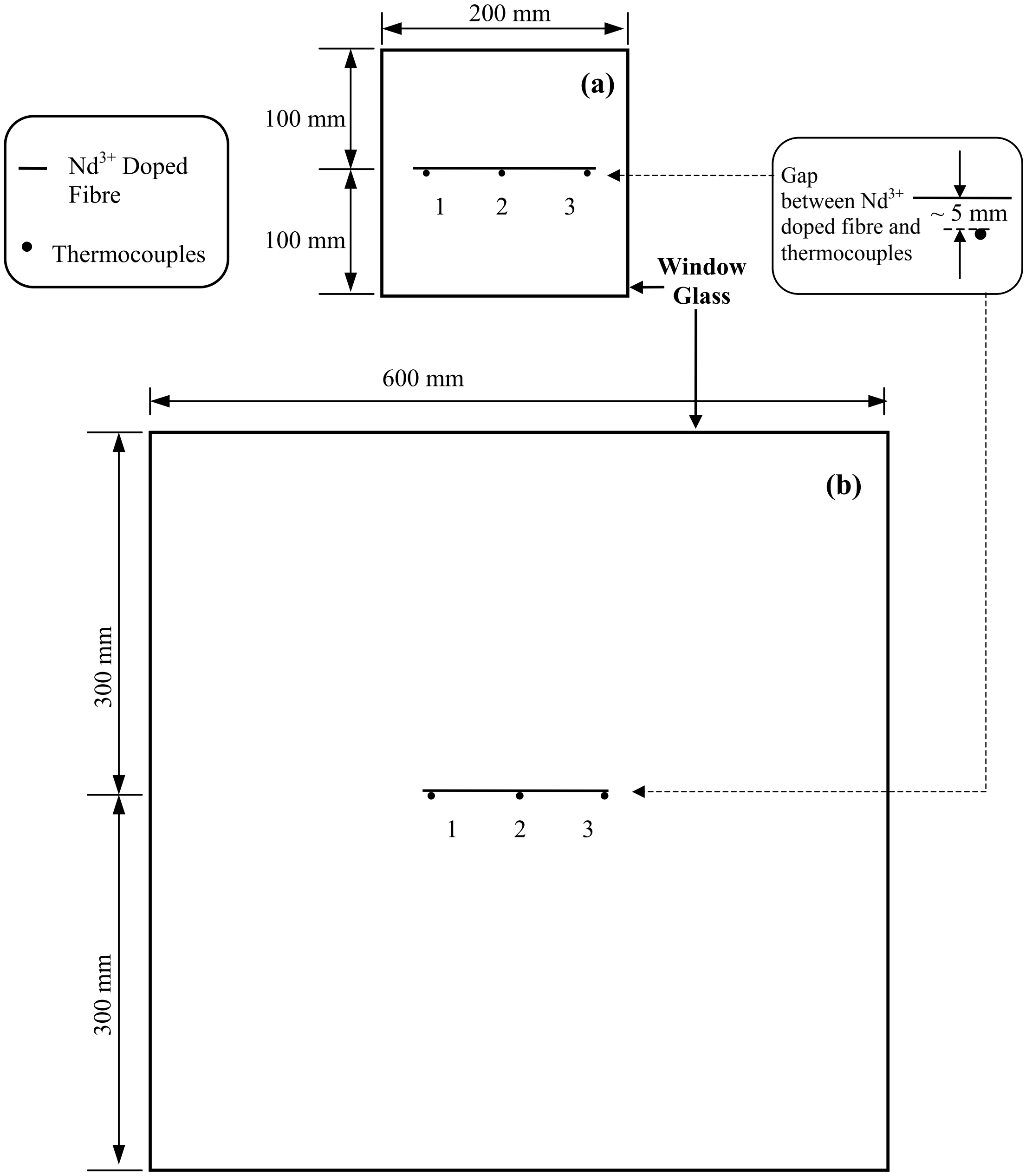


Figure 4.4.1 The placement of thermocouples (denoted by 1, 2 and 3) and neodymium doped fibre of length 150 mm attached to the centre of (a) the small windows (used in the fire simulation tests 2 and 3) and (b) the larger window as used in tests 4 and 6. Similar configurations were used for different lengths of doped fibre used in other fire simulations (see Table 4.4.1).

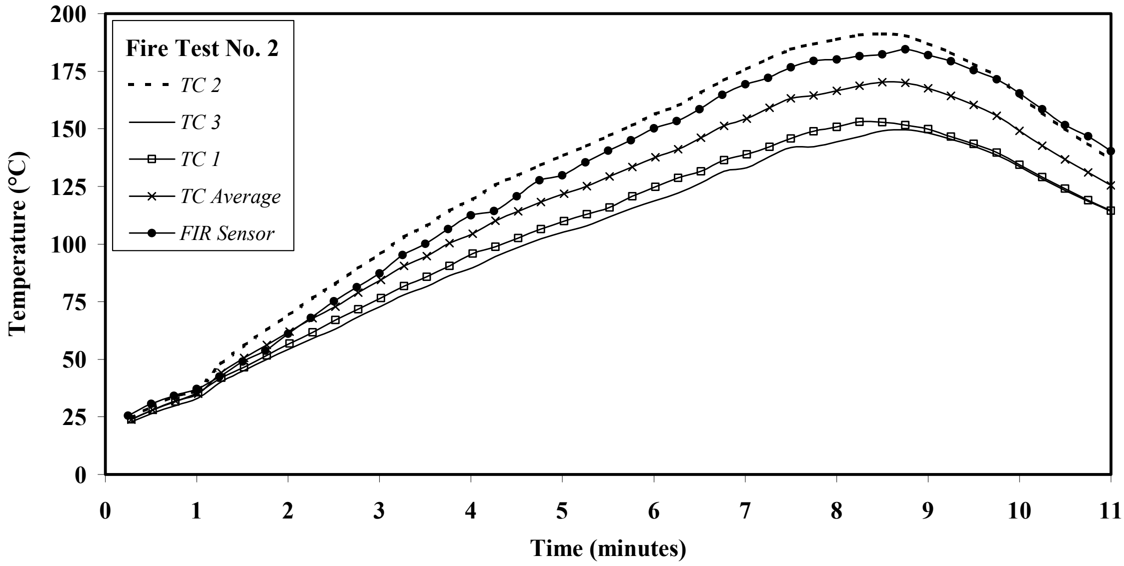
Table 4.4.1 Summary of the fire simulation tests using the fluorescence intensity ratio sensor to measure window glass temperature.

Fire Simulation Test N$^{o.}$	Nd^{3+} doped Fibre Length (mm)	Temperature Range of Calibration (°C)	Window Dimensions (mm x mm x mm)	Adhesive used to Attach the Fibre to the Glass	**Notes** Time of window cracking is given in minutes (') seconds (").
1	180	40 – 240	200 x 200 x 3	*KS Bond* super glue	Adhesive applied along the length of the fibre. Temperature measured every 15 seconds. The test was a trial of the performance of the fibre sensor in a fire and the adhesive used for fibre attachment. FIR sensor was successful in measuring the temperature of glass in a fire. FIR sensor followed the trend of the thermocouple temperatures. Window did not fracture.
2	150	20 – 230	200 x 200 x 3	*KS Bond* super glue	Adhesive applied along the length of the fibre and cured for 1 hour. Temperature measured every 15 seconds. Window did not fracture.
3	150	20 – 250	200 x 200 x 3	*KS Bond* super glue	Adhesive applied along the length of the fibre and cured for 1 hour. Temperature measured every 15 seconds. Window did not fracture.
4	150	20 - 240	600 x 600 x 6	*KS Bond* super glue	Adhesive applied along the length of the fibre and cured for 1 hour. Temperature measured every 15 seconds. Thermocouple 2 detached during the test. Window cracked at about 6'20".
5	180	20 - 160	600 x 600 x 3	*KS Bond* super glue	Adhesive applied along the length of the fibre and cured for 1 hour. Temperature measured every 15 seconds. 1st window fractured at 3'35" and 2nd window fractured at 5'25". Fuel exhausted at 10'.
6	150	10 – 210	600 x 600 x 3	*KS Bond* super glue	Adhesive applied at points along the fibre. 1st window fractured at 3'35" and 2nd window fractured at 6'. Fuel exhausted at 10'.
7	200	20 – 190	600 x 600 x 3	*KS Bond* super glue	Adhesive applied at points along the fibre. 1st window fractured at 3'55" and 2nd window fractured at 6' 30" Fuel exhausted at 10'. Glass edges shaded with millboard (40 mm width).
8	155	20 – 180	600 x 600 x 3	cement	Adhesive applied at points along the fibre. Fuel exhausted at 9' 45". 1st window fractured at 3'24" and 2nd window fracture at time unknown. Edges of glass shaded with millboard (40 mm width).

4.4.2 Measurement of Glass Temperature

A summary of the fire simulation tests of the windows is given in Table 4.4.1. The description includes the length of the fibre probe, the temperature range of calibration of the fibre sensor, the adhesive utilised to attach the fibre to the window, and the thickness of the glass. Figure 4.4.2 (a) and (b) illustrate the results from the compartment fire simulations using the smaller sized windowpanes for tests 2 and 3, respectively. The results of test 1 are not shown as the doped fibre probe was incorrectly positioned with respect to the configuration of the thermocouples. The test was viewed as a trial of the measurement capability of the FIR sensor during fire conditions and of the adhesive used for attachment of fibre to window glass. Figure 4.4.3 (a) and (b) show the results of fire simulation tests 4 and 5 respectively, in which larger sized windowpanes (600 mm x 600 mm) were used. Figure 4.4.4 (a) and (b) show the results of fire simulation tests 6 and 7 respectively. The outcome of test number 8 is shown in Figure 4.4.5.

(a)



(b)

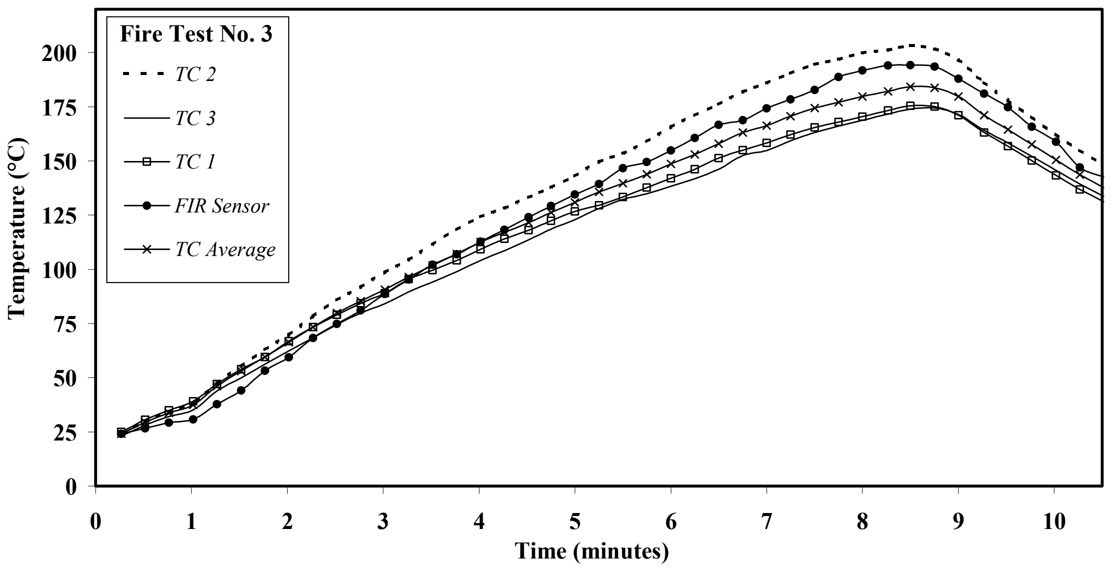
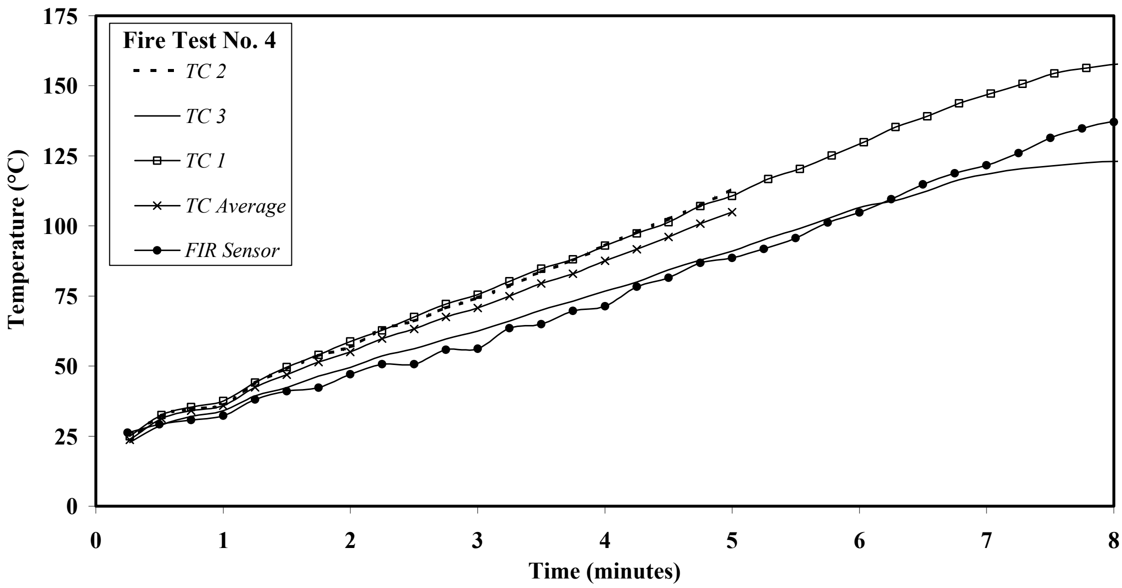


Figure 4.4.2 Measurement of small window glass (300 mm x 300 mm) temperature using the FIR sensor and thermocouples during fire simulation tests 2 and 3, shown by (a) and (b) respectively. (TC = thermocouple.)

(a)



(b)

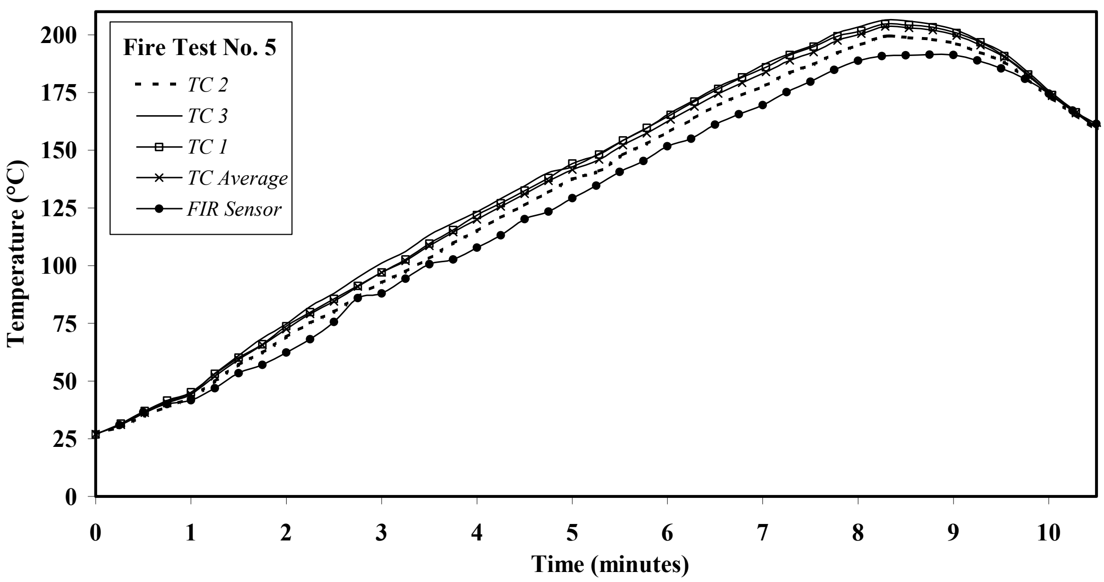
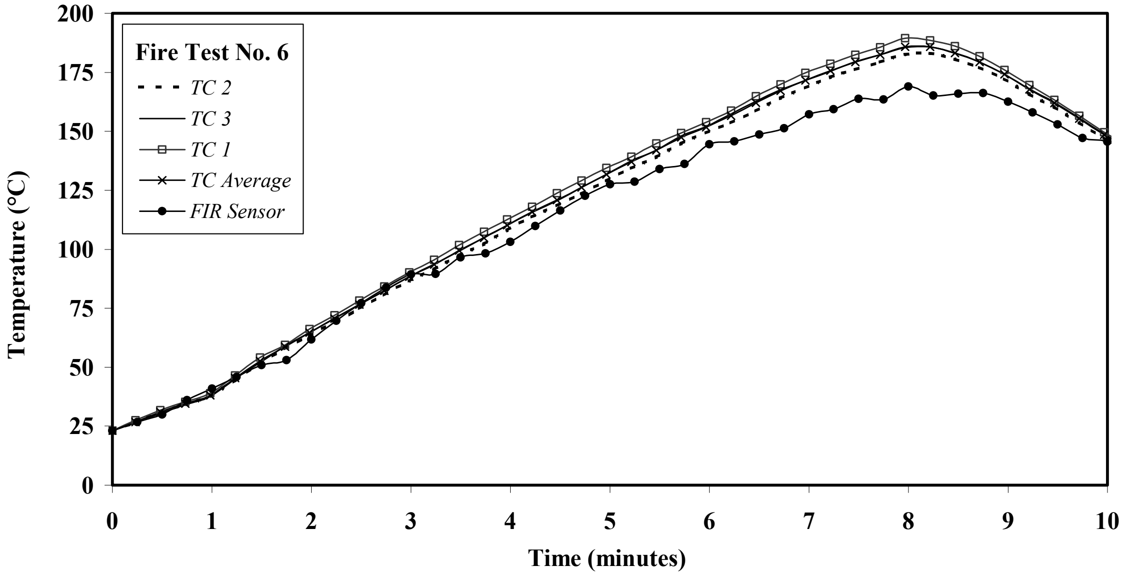


Figure 4.4.3 Measurement of large windowpanes (600 mm x 600 mm) with the FIR sensor and thermocouples during fire simulation tests 4 and 5, shown by (a) and (b) respectively. (TC = thermocouple.)

(a)



(b)

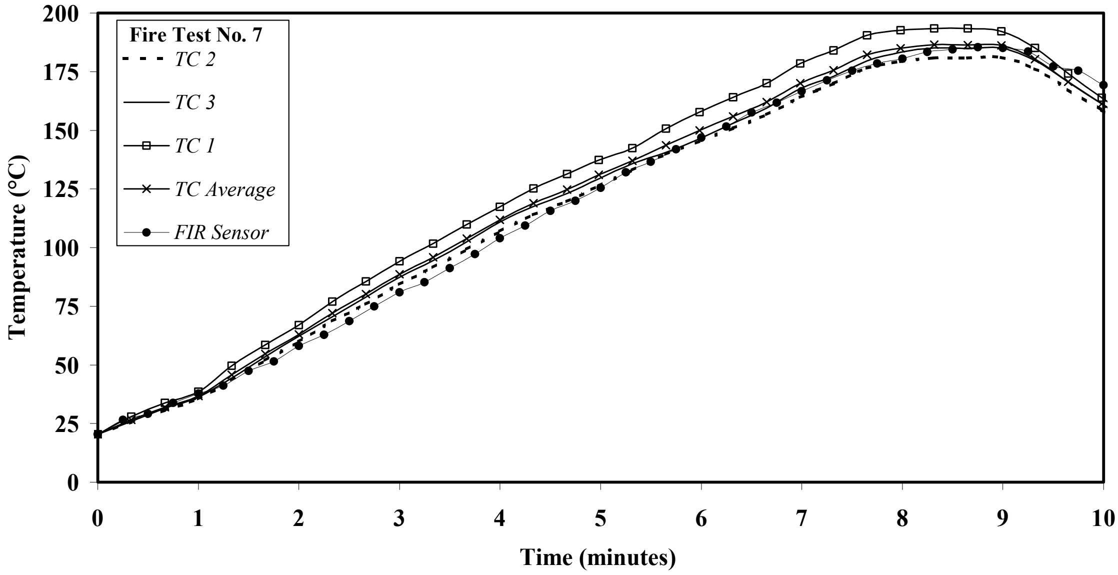


Figure 4.4.4 Measurement of large window glass (600 mm x 600 mm) temperature using the FIR sensor and thermocouples during fire simulation tests 6 and 7, shown by (a) and (b) respectively. (TC = thermocouple.)

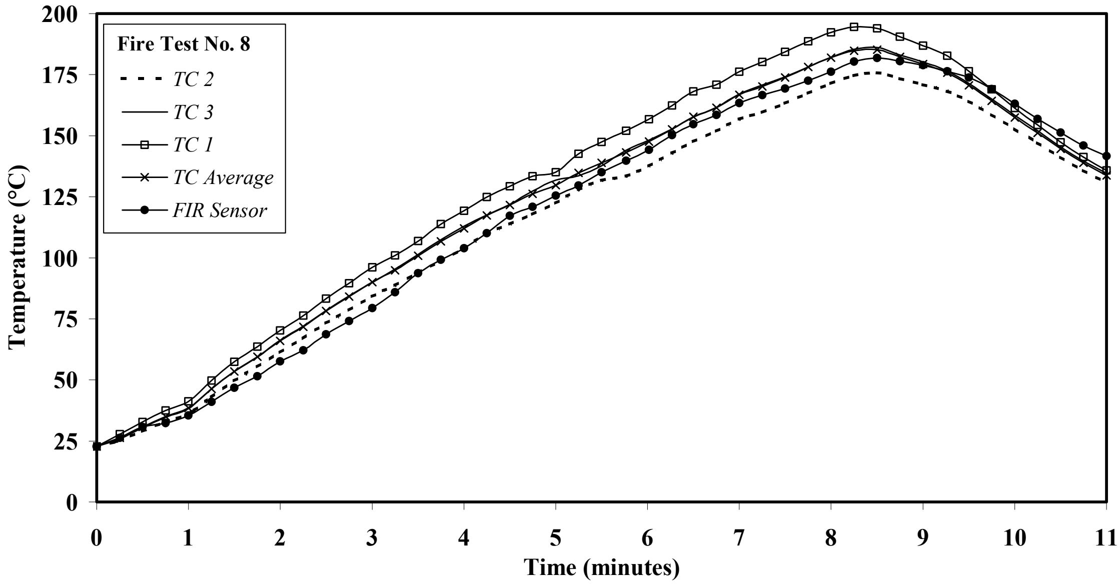


Figure 4.4.5 Measurement of large window glass (600 mm x 600 mm) temperature using the FIR sensor and thermocouples during fire simulation test 8. (TC = thermocouple.)

4.4.3 Discussion of Measurement of Glass Temperature During Fires using the Fluorescence Intensity Ratio Sensor

Small Windows

In the first fire simulation (Test 1 in Table 4.4.1), a small window (with dimensions 200 mm x 200 mm x 3 mm) was used. The test was a trial of the performance of the fibre sensor and the fibre adhesive in a fire. The fluorescence intensity ratio sensor operated successfully during the fire test. The doped fibre was undamaged by the fire and the *KS Bond* glue retained its original colour. On the whole, the temperatures measured by the fibre sensor followed a similar temperature trend as the thermocouples, comparable to the trend shown by the other fire test results.

Windows (with dimensions 200 mm x 200 mm x 3 mm) were again used for tests 2 and 3. The length of neodymium doped fibre was 150 mm for both of these tests. *KS Bond* adhesive was used along the length of fibre to attach the sensor to the window. The FIR sensor and the thermocouples follow the same temperature trend for the two tests. As illustrated in Figure 4.4.2, the centre-positioned thermocouple 2 (see Figure 4.4.1 for its arrangement) measures the highest temperatures. The doped fibre temperature is higher than the average temperature of thermocouples 1, 2 and 3. The glass did not fracture, most likely due to the small size of the glass (heating more evenly than the larger panes) and insufficient temperature gradients.

In the bench scale radiant heat tests that were designed to simulate radiation present in a fire (as described in Section 4.3), a distinct discrepancy between the temperature measured by bare fibre and bare thermocouples was demonstrated (see Figure 4.3.2).

The advantage held by the bare doped fibre sensor of being less susceptible to heat radiation than thermocouples was diminished by the application of an adhesive to the doped fibre probe. The fibre coated in *KS Bond* revealed consistent temperature trends relative to the thermocouples when subjected to this same radiant heat (see Figure 4.3.3 (a)). In the case where the *KS Bond* coated fibre and bare thermocouples are exposed to the heat radiation from a fire, similar results as that seen in Figure 4.3.3 (a), of consistent temperature trends of the probes, could be expected. However, for the fire tests 2 and 3, the *KS Bond* coated fibre shows higher (though consistent) temperature trends than that of the thermocouples (average) temperature. The variation in this set up is the attachment of the thermocouples to window glass using *Pyropanel* acrylic.

The higher temperatures measured by the centre-positioned thermocouple 2, compared to the temperatures measured by thermocouples 1 and 3 which were closer to the window edges (see Figure 4.4.1), indicate that it is likely that sections of the doped fibre probe were also experiencing differing temperatures. This is particularly the case as the length of fibre almost spanned the width of the small window glass. The middle of the doped fibre section, which was close to thermocouple 2, would have been at a higher temperature than the ends of the fibre that were next to thermocouples 1 and 3. These latter thermocouples were near the edge of the windowpane and kept at a lower temperature by the mounting frame of the window. It is also considered that since the fluorescence in the doped fibre is counter propagating, one end of the fibre may produce a stronger fluorescence intensity than other parts of the fibre. The radiation effects of the fire impact on the thermocouples and fibre differently. These radiation effects, in addition to the issues discussed above, may have contributed to the higher temperatures measured by the fibre than those indicated by the thermocouple average temperature.

Large Windows

The large windows used in tests 4 to 8 were 600 mm x 600 mm in size. The thickness of the window used in test 4 was 6 mm. For fire tests 5 to 8, windows of thickness 3 mm were used. This was required for the complementary research work so as to induce fractures in the window more quickly. The various neodymium doped fibre lengths used in each fire test are as listed in Table 4.4.1. During fire test 4, thermocouple 2 detached from the window at about 5 minutes. Data for this thermocouple and the average thermocouple temperature are shown (in Figure 4.4.3 (a)) only up to this time.

The fibre adhesive used was *KS Bond* super glue, except in test 8, where the same adhesive used for the thermocouples was also used for the fibre, the adhesive being an acrylic sealant. Applying the adhesive along the length of fibre might result in some adhesive coming in between the fibre and glass. In tests 6, 7 and 8 the adhesive was applied to points along the length of fibre while ensuring that the fibre was in contact with the glass. This was to increase the surface contact between the fibre and window. It was also an attempt to leave as much of the fibre probe bare as possible, since the bench scale radiant heat tests (described in section 4.3) showed bare fibre to have a distinct advantage over the thermocouples and their susceptibility to radiation. For the fire tests, it is difficult to quantify the exact change in the fibre temperature trends where the adhesive has been applied along the length of the fibre or merely at points. This is since each test had varied parameters; the widths of the windows were different in test 4 and 5, and the length of the fibre probe differed in tests 5 and 6 (see Table 4.4.1). However, similar trends have been demonstrated for the two methods of application as shown by tests 5 and 6 (Figures 4.4.3 (b) and 4.4.4 (a) respectively).

For fire tests 4 to 8, the centrally located thermocouple 2 demonstrates the lowest temperature trend of the thermocouples, except in the case of test 4 (Figure 4.4.3 (a)) where the thickness of the window was 6 mm as opposed to 3 mm for the other tests. Also in test 4, thermocouple 3 seems to follow an inconsistent temperature trend toward the end of the test, although it is suspected that the thermocouple partially detached from the window at that stage. The thermocouples in all of the other fire tests (Figures 4.4.3 (b), 4.4.4 and 4.4.5) demonstrate consistent temperature trends for the duration of the fire simulations.

Unlike for the small windows, the FIR fibre sensor demonstrates markedly lower temperatures than the (average) thermocouple temperature during tests 4, 5 and 6 (as seen in Figures 4.4.3 (a), 4.4.3 (b) and 4.4.4 (a) respectively). The difference between the fibre sensor and the thermocouples is approximately 10 ºC starting from about 3 minutes into the test, to about the time the temperature has peaked. It is during this time that the fire is significantly growing in intensity and during which radiation from the flames and hot gases are most prevalent.

The results from fire tests 7 and 8 (illustrated by Figures 4.4.4 (b) and 4.4.5 respectively) indicate that the FIR fibre sensor is generally still showing a lower temperature than the thermocouple average (the difference between them being less than 10 ºC). However, the discrepancy is not as marked as that for tests 4, 5 and 6. A possibility for this is the shading depth used on the windows. In all of the fire simulations, a frame (of depth 10 mm) shaded the edges of the windows. The frame enabled the mounting of the window to the wall. In tests 7 and 8, the window edges were additionally shaded with millboard. Additional fire simulations conducted by Iraida Khanina (in which the FIR sensor was not used) demonstrated the consequence of shading and framing on window breakage. In similar test conditions, the cases where the entire window (neither framed, nor additionally shaded) was subjected to the fire, it did not crack. A relationship between the shading dimensions used and the breakage time of windows was not established Khanina *et al* [2000]. However, its significance is recognised. The additional shading of the panes in tests 7 and 8 might impact the temperature difference between the centre of the pane and the edge, as well as the temperature gradient along the length of the fibre. These effects may manifest as fibre temperatures that are higher than those demonstrated in tests 4, 5 and 6.

In the early stages of the fire simulation tests (about 1 to 3 minutes) the deviation in the temperatures measured by the FIR sensor and the thermocouples start to become more pronounced. This tendency continues as the fire grows and reaches its maximum intensity during which the heating process is largely radiative. After the maximum temperatures in the chamber are reached and approaching the time that the fuel is about to be depleted, the divergence in temperatures measured by the FIR sensor and the thermocouples is decreased. This is likely due to the decrease in the radiative heating processes occurring in the chamber. In Tests 6, 7, and 8, the adhesive was applied only along points on the fibre to minimise the impact of the adhesive. For these tests in particular, the divergence of temperatures during the heating stage, demonstrate that the impact of radiative heating effects on the temperatures measured by thermocouples is significant. In the absence of radiant heat, the convergence of temperatures may have been further demonstrated had the recording of temperatures continued for an extended time even after the fuel had been exhausted. This additional data would have been helpful and possibly supported the confidence in the stability of the FIR system.

The thermal conductivity of the thermocouple is considerably higher than the thermal conductivity of the fibre sensor. Since both of these sensors are attached to the glass, the conductivity of the glue and its effect on the thermocouples and fibre is of interest.

Typical total emissivity for glass is 0.95 (at 300 K). Data is not available for nickel based alloys of K-type thermocouples, however the typical total emissivity for oxidised nickel is 0.4 (at 300 K). The total emissivity of aluminium and chromium are typically within these values [Brewster, 1992]. More importantly, the emissivity of glass is significantly higher than that of the metal based thermocouple. That is, radiation emitted by glass is significantly higher than the radiation emitted by thermocouples. This implies that the heat transfer is largely radiative, since thermocouples are more susceptible to radiation than the fibre sensor.

The bench scale tests showed a much higher discrepancy (about 25 ºC for thermocouple temperatures greater than 100 ºC) between the fibre sensor and thermocouples than that demonstrated in the fire simulations. For both types of tests, the temperatures of each of the probes deviate after about 2 minutes of heating time. For the bench scale tests, the applied radiation was from a household bar heater that was placed in front of the fibre and thermocouples. Although the heat from the bar heater was intended to simulate radiation generated by a fire, the heat radiation from the household heater may have been greater than that produced in the fires. Additionally, the applied heat from the bar heater was consistent right up to the time at which the heater was switched off, whereas the dynamics of a fire are variable. Also, during the fires, the thermal mass of the window itself may have slowed the temperature rise of the fibre compared to the lone fibre of the bench scale tests.

The bench scale tests showed *KS Bond* coated fibre to follow similar temperature trends to that of bare thermocouples. Since in the fire tests, the thermocouples are attached to the windows with an adhesive, this adhesive possibly influences the thermocouple measurement in some way. It is useful to note that the application of adhesive is not reproducible neither for the attachment of the thermocouples nor the attachment of the fibre to the window glass. Thus quantitative measurement of the effects of the adhesive cannot be made. Therefore, its impact can only be described in general terms. Although the fibre sensor and thermocouple are affixed to the fibre using unlike substances, the fibre sensor is still measuring a lower temperature suggesting that fibre is less susceptible to heat radiation than thermocouples. For the latter three large scale fire simulation tests, in which minimal adhesive was applied, the radiation effects on thermocouples was demonstrated to be significant as discussed above.

Chapter 5

Conclusion

5.1 INTRODUCTION

The aim of this research was to employ a prototype optical fibre temperature sensor to measure the surface temperature of window glass during the simulation of small-room fires. The fibre sensor was based on the fluorescence intensity ratio (FIR) technique [Baxter *et al*, 1996]. This technique utilises fluorescence from a suitable pair of energy levels of rare earth doped optical fibres. The particular device used neodymium doped fibre as the temperature measuring probe. The sensor was a variation on that reported by Muscat [1997] and Wade *et al* [1999]. The advantages of optical fibre sensors, in general, over other temperature sensing tools are that they are small in size and weight, and can operate in electromagnetically affected environments. The fluorescence intensity ratio method has proven to be an important temperature sensing technique [Wade, 1999]. It is relatively low in cost, simple in configuration, and practically insensitive to strain [Wade *et al*, 2000a]. The versatility of the fluorescence intensity ratio method is being demonstrated with a dual strain and temperature sensor being the focus of current researchers [Trpkovski *et al*, 2003].

Fires are an important area of research because of their potential danger to human life and property. Since the breakage of windows can significantly increase the tragedy caused by a fire, the role of window glass is an important one [Emmons, 1986]. Measurement of the temperature of windows is usually with thermocouples. Unfortunately, the thermal properties of windows and thermocouples are dissimilar [Luo, 1997]. As such, there is discrepancy in experimental data and theoretical models of window breakage [Keski-Rahkonen, 1988; Skelly *et al,* 1991; Joshi and Pagni, 1994a]. The inconsistency is ascribed to radiative heating of thermocouples, and the inability to discriminate the true temperature from radiation effects [Janssens and Tran, 1992].

The work of this thesis endeavoured to measure the windows with a better matching sensing device, the fluorescence intensity ratio method optical fibre sensor. The similar thermal properties of windows and optical fibre meant that a closer to genuine temperature was achieved by use of the sensor. In addition, the other advantages of the fluorescence intensity ratio method qualified its suitability for this application. Commercial temperature sensors could not be used because of their expense and the destructive aspect of the fire simulations. The window and the sensor probe were sometimes destroyed during the fire tests. A new section of neodymium doped fibre was used for each test. The low cost of the fibres meant that this could be achieved easily. The relatively slow response time of distributed sensors meant they were unsuitable for the dynamic nature of a fire. Fluorescence intensity ratio sensors can have short (a few centimetres) sensing probes meaning better spatial resolution and a faster response time. As the window surface may undergo strain during the fires, the strain insensitivity of FIR systems made them more suitable than other fluorescence based techniques that do not have a near zero strain sensitivity.

5.2 APPLICATION OF FLUORESCENCE INTENSITY RATIO SENSOR FOR MEASUREMENT OF WINDOW GLASS TEMPERATURE

To prepare the fluorescence intensity ratio sensor for measurement of window glass temperature during fires, an assessment of suitable adhesives for attaching the fibre and thermocouples to the glass was done. Following this, the operation of the fibre sensor and thermocouples were compared as they were exposed to radiant heat. The fluorescence intensity ratio sensor was then used for measurements of window glass temperature in an experimental fire chamber.

The neodymium doped sensing fibre was typically of doping concentration 900 ppm, had a core diameter of 5 μm, and was 150 mm in length. The spatial resolution of the FIR sensor would have improved from a shorter doped fibre section. This was not done since the consequence of the fibre being shortened further is a reduction in fluorescence intensity, particularly at the smaller wavelength. Difficulties would then be encountered with discerning this signal from noise. The coating on the fibre was removed to eliminate any effect of the coating on temperature measurement and to enable optimal fibre-to-glass contact. Neodymium doped fibre was fusion spliced to the FIR sensor. The sensor was calibrated usually from room temperature (~ 20 ºC) to a maximum of 250 ºC, which was sufficient for window temperature measurement in the fire simulations. The fire simulations were scheduled to closely follow the calibration in order to limit stability issues with the laser diode pump source. The calibration data was fitted to a fourth order polynomial, and a reference or 'look up' table was generated for the actual fire test. The standard deviation between the experimental data and the fit was 1.4 ºC, with the maximum deviation being 5.4 ºC.

Five different adhesives were assessed for their suitability for attaching the fibre to the window glass. Other considerations included their ability to withstand a fire environment, their ease of application, curing time, operating temperature, and their appearance when exposed to radiant heat, in particular flames. Domestic super glue named *KS Bond* was selected, as it met the above requirements.

Bench scale tests to compare the performance of the fibre sensor and thermocouples when subjected to radiant heat were performed. A household bar heater was used to simulate the radiation present in a fire. For thermocouple temperatures above 100 ºC, bare neodymium doped fibre measured temperatures approximately 25 ºC less than the thermocouples. This discrepancy was an indication of the effect of radiative heating on thermocouples. It was consistent with the understanding that the fibre sensor, being less affected by radiation, would have an advantage over thermocouples during fire tests. However, this advantage would be lost with the application of an adhesive to the doped fibre, which was necessary for the attachment of the fibre to window glass. Comparative tests of the fibre sensor coated in *KS Bond* and bare thermocouples showed the trend of the fibre temperature to closely follow those of the thermocouples.

The fluorescence intensity ratio sensor was used to measure the temperature of window glass in eight fire experiments. A specially designed chamber (for the work of Iraida Khanina) was constructed to simulate a fire in a small room and conditioned to induce window breakage. Investigations of the mechanisms of window glass breakage [Khanina *et al*, 2000] were simultaneously carried out during the fire simulations, which are about 10 minutes in length.

Two windowpane sizes were used. The smaller windows were 200 mm x 200 mm x 3 mm (width). For the fire tests, the thermocouples were attached to the glass with *Pyropanel*, a fire rated acrylic sealing compound. The doped fibre section was adhered to the window using *KS Bond* super glue applied along the length of fibre. The fire tests using these small windows show the FIR sensor to follow the same temperature trend as the thermocouples, but with higher temperatures than the thermocouple average.

The dimensions of the large windows were 600 mm x 600 mm. Applying the adhesive along the entire length of the fibre, increased the possibility of a thin layer of adhesive coming in between the fibre and window. For the latter three of these fire tests, the technique for attaching the doped fibre probe to the window was improved. This aimed to leave the fibre probe as bare as possible and retain the advantage of bare fibre over thermocouples in being less affected by radiation, as was shown in the bench scale radiation heat tests. The adhesive was applied at a minimum number of points along the fibre. It was ensured that the fibre and glass were in contact. For these tests in particular, the deviation of temperatures during the heating stage where radiative processes are dominating, demonstrate that the influence of these effects on the temperatures measured by thermocouples is significant.

In each fire test, a window was mounted to the chamber via a frame, which shaded the edges of the window. In two of the tests, the windows were additionally shaded with millboard. For the fire tests in which the windows were not additionally shaded, the FIR sensor measures a lower window glass temperature than the thermocouples. The difference is approximately 10 ºC less than the average thermocouple temperature for the majority of the fires. In the fire tests where the windows are shaded by millboard, the FIR sensor is generally measuring a lower temperature than the thermocouple average, with the difference not as pronounced. Khanina *et al* [2000] did not correlate shading depth with the time of window breakage. However, Khanina *et al* reported that unframed and unshaded windows remained intact during similar fire simulations. It may be possible that extra shading impacts the temperature difference between the centre of the window and the edge, as well as the temperature gradient along the length of fibre. For the length of neodymium doped fibre used, this might have been realised as higher fibre temperatures. Nevertheless, the fibre sensor generally measured lower temperatures than the thermocouples during the fires.

Overall, the work of this research demonstrated the successful application of the fluorescence intensity ratio sensor for the investigation of window surface temperature during fires. Further, a discrepancy between the effect of thermocouples and the fibre sensor to a radiant heat environment was clearly established.

5.3 FUTURE WORK

There is a place for optical fibre temperature sensors in fire research, especially if the system is low in cost and capable of multiplexed measurements. The fluorescence intensity ratio method was successfully used to measure windows during fires. Although for the prototype sensor used in this work, significant limitations on its practicability were present. Improvements to the sensor could be made by the addressing of issues such as laser diode stability and the signal to noise ratio in the detectors. Annealing the doped fibre probe would additionally improve the reliability and repeatability of the FIR sensor. Further, the window measurement testing procedure would benefit from the use of a shorter length of doped fibre (about 1-2 cm). The shorter length would ensure a better real-time response and enable improved spatial resolution of the temperature measurement. A shorter fibre is possible if the doping concentration of the fibre was suitably higher. However, for window glass temperature measurement, other fluorescence based techniques may be more convenient than optical fibre sensors.

The challenge facing all contact temperature sensors is the matching of the probe to window glass to ensure a faithful measurement of window glass temperature. A genuine representation of window temperature may be achieved by a remote sensing system, or one that minimises contact with the glass. A potential technique is the use of the temperature dependent ratio between Stokes and anti-Stokes wavelengths from Raman effects. The remote measurements could be made by the employment of optical fibre probes. To be viable, this distributed sensing system would need to have an adequately fast response time. Further, any sensing system to be considered should be low in cost and uncomplicated in design. One potential system for utilising fluorescence based sensing methods is the application of a thin film on the window surface. The thin film would be some sort of rare earth doped material. To obtain the fluorescence intensity ratio, two charge-coupled-device (or CCD) cameras could be used to measure the fluorescence intensity (filtered at appropriate wavelengths). Background radiation effects would need to be quantified. Alternatively, a pulsed laser (for example, every 5 seconds) could be used in a fluorescence lifetime based sensor, enabling temperature measurement of any part of the windowpane with a single detector. The fast response time of fluorescence decay time sensors makes them suited to the frequently changing fire environment; strain effects would need consideration, however, depending on the section of the window being measured. For both fluorescence methods, the effects of the thin coating on the window glass surface on the behaviour of the glass would need to be taken into account.

5.4 CONCLUSION

Overall, the fluorescence intensity ratio method has demonstrated the measurement of temperature in a fire environment. The work from this thesis has contributed to a better understanding of the performance of a prototype sensor. This work is also useful in the consideration of fluorescence based techniques to be applied to window surface temperature measurement.

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