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1*	Balazs Ihracska	University of Hertfordshire, School of Engineering and Technology, College Lane Hatfield, Hertfordshire AL10 9AB, UK	a.b.ihracska@herts.ac.uk
2	Roy James Crookes	School of Engineering and Materials Science, Queen Mary University of London, London E1 4NS, UK	r.j.crookes@qmul.ac.uk
3	Diogo Montalvão	Department of Design and Engineering, Faculty of Science and Technology, Bournemouth University, Talbot Campus, Fern Barrow, Poole, Dorset BH12 5BB, UK	dmontalvao@bournemouth.ac.uk
4	Mohammad Reza Herfatmanesh	University of Hertfordshire, School of Engineering and Technology, College Lane Hatfield, Hertfordshire AL10 9AB, UK	m.r.herfatmanesh@herts.ac.uk
5	Zhijun Peng	University of Hertfordshire, School of Engineering and Technology, College Lane Hatfield, Hertfordshire AL10 9AB, UK	z.peng2@herts.ac.uk
6	Shahid Imran	Department of Mechanical Engineering (KSK Campus), University of Engineering and Technology, Lahore, Pakistan	shahidimran 512@hotmail.com
7	Theodosios Korakianitis	Parks College of Engineering, Aviation and Technology, Saint Louis University, St. Louis, MO 63103, USA	talexander@slu.edu

\*corresponding author

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# Abstract

9 In this study, the design aspects of optically accessible pressure vessels are investigated via a case study of a High Pressure 10 Combustor experimental rig. The rig was designed to take optical measurements of combustion, simulating the conditions 11 found in internal combustion engines and turbines. Although, it is not new to equip chambers and reactors with sight windows, important aspects of design and relevant information regarding optical access is missing or are insufficiently 12 13 explored or not readily accessible in the existing literature. A comprehensive review of requirements for optical access to 14 such high-pressure, high-temperature systems has been conducted. It is shown in a readily-navigable format as function of 15 application and precision, with data and technical correlations hitherto not found in a 'user-friendly' style. The material 16 selection procedure is detailed and supported by a complete comparison of optical materials and relevant properties. The review revealed a significant inconsistency in mechanical properties claimed in the literature for optical materials. As a 17 18 response to this, increased safety factor values are suggested as function of level of uncertainties and effects of failure, typically three to four times higher than the industrial standard. Moreover, newly developed equations are presented linking 19 20 performance analysis to the design criteria.

21

22 Keywords:

- Optically-accessible reactor,
- Optical engine,
- Pressure vessel;
- Sapphire,
- Window design,
- 28 Combustion

# 29 1 Introduction to the High Pressure Combustor (HPC) and the need for optical development

Over the past two decades, concerns about global warming and the depletion of the ozone layer have driven researchers to find better alternatives to the high energy consumption demand [1-4]. With the combustion of fossil fuel and the subsequent production of carbon dioxide being accounted for as the main contributor to the current release of greenhouse gases to the atmosphere [5], and taking into consideration that a solution to the current energy supply problems is yet distant, improvements in the understanding of the chemical reaction and flame-propagation processes and reduction the emissions of these engine-fuel combinations should be implemented as a short term solution [6-8].

36 The HPC was developed to address research topics in combustion science. Its unique design makes it a versatile tool to 37 model and test the working, at real-life conditions of industrial furnaces, external and internal combustion engines and gas 38 turbines. It can be set up to test steady combustion up to 60 bar for 30 minutes. It can accept virtually any combustible 39 substance with a high accuracy of air-fuel ratio and a control of residence time. Moreover, the flow pattern can be set to 40 either plug or swirl. The high-pressure air (variable up to 60 bar) is delivered to the chamber via a number of safety 41 instruments from a large air receiver – which is charged by a three-stage piston pump. The air arriving to the combustion 42 chamber is dried, and its flow and pressure is set by a computer-controlled valve system. The fuel is injected into the 43 chamber by interchangeable injectors; the fuel flow pattern, supply pressure and volume flow is variable. The actual 44 combustion chamber is not a single-piece vessel but rather an assembly of several sections. Therefore, the length of the 45 chamber – and hence, the residence time of reactants – can be varied depending on the application. The sections were 46 designed with numerous radial access points so that reaching any point inside the combustor for sampling would be 47 possible; see Figure 1 for a schematic of the experimental rig. The initial ignition is provided by a high-energy spark. At the 48 end of the process, the burned mixture leaves the chamber via a special plunger valve that is capable of withstanding the high temperature and pressure. A detailed description of the HPC facility can be found in [9-11]. 49



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Figure 1. Schematic drawing of the experimental rig, adapted from [10].

The HPC has supplied the combustion research community with vital information. Using thermocouples and sample probes, its current capabilities have been fully utilised. New developments were needed to keep the rig up-to-date. In the last 15 years or so, the optical- and laser-based measurements became the most important tools to investigate combustion details and results of these methods published elsewhere [12-14]. These methods have a fast response time and do not require actual physical contact with the flames. It was essential to equip the HPC with optical access in order to keep the research work current and on-going.

## 58 2 Current contribution

59 The design methods and procedures of industrial pressure vessels are well-documented, with comparisons of methods and 60 standards available for industrial applications [15-25]. These provide good guidelines even for an unconventional design 61 task, but of course they do not provide comprehensive data for all possible cases. In general, the available literature lacks data regarding optical access to pressurised vessels. Information on design practices and material properties are scattered
 in the literature, being hard to find and often inconsistent. Therefore, the aim of this work is to target these gaps by:
 collecting the scattered data; dealing with inconsistency found in literature and providing detailed recommendation for
 safety factors, collecting and presenting design criteria as function of application; developing new functions and equations.

In this work, novel complementary material is provided for the design of chambers and reactors that require the equipment 66 67 of sight windows on them. As a result of extensive review, the properties of practical optical materials were collected and 68 presented together in graphs and tables, allowing for direct comparison. The details of special design practices regarding 69 transparent parts are discussed, and the available data on existing design solutions is collected and shown. Some 70 complementary material is added to the basic equations and relations in Statics. Moreover, there are papers examining 71 design procedures [26], but to the authors' knowledge this is the first detailed design study on optically accessible pressure 72 vessels (fixed volume or internal combustion optical engine) where the real-life application of the collected data is shown. 73 Structural analysis of optical material window is shown and its effects to practical design.

# The optical access: review of material and their properties; practical solutions; mechanical and optical performance



76 **3.1** Optical materials: mechanical, optical and chemical properties

77

Figure 2 Transmittance of the reviewed optical materials; relative responsivity and wavelength of interest are also
 shown (for Design requirements section).

There are a large number of materials that can be considered for sight windows on pressure vessels, from ordinary plastics to exotic ceramics. In this work, only the most common and most practical optical materials were chosen for comparison.

The underlying design criteria for selecting the optimal optical material type are: useful transmittance range, operating temperature and mechanical load. In Table 1, only high-operating-temperature materials are listed. It is important to note there are other choices available for specialised tasks, such as silicon or germanium, but their availability is limited and they are costlier. In low-temperature environments, plastics like acrylic and polycarbonate can be used. During design, it is essential to consider the working temperature and obtain a good estimate of it from simulations or experiments.

87 Although unusual in mechanical engineering, it is important to choose the right material for the required electromagnetic 88 band. It is also vital to consider the ratio of the electromagnetic energy falling on a body to that transmitted through it. This 89 ratio is called the transmittance of the material [27]. Transmittance values for each wavelength vary significantly among 90 material. For example, a larger selection of materials can be considered if the investigated radiation is in the visible or in the 91 near infra-red (NIR) regions. Due to the availability of a wider range of materials, the implication is that sight windows for 92 high speed imaging or laser-aided measurements can be designed more easily, and more complex shapes with larger 93 dimensions are therefore possible. Choosing an optimal material is more complex when longer wavelengths have to be 94 captured for both spectroscopy and thermal imaging. For wavelengths over 2500 nm, the transmittance curves start 95 fluctuating or becoming discontinuous. If this, then, is the electromagnetic wave band region of interest, careful planning 96 will be needed to select the right material type. The transmittance of common optical materials for wavelengths under 200-97 250 nm falls rapidly. Yet, it is an important region in combustion science as some radicals have their peak emissivity in this

- 98 electromagnetic band. Researchers and designers are practically left with fused silica and a number of fluorides (MgF<sub>2</sub>, CaF<sub>2</sub>,
   99 BaF<sub>2</sub>) to use. Figure 2 shows the transmittance curves of selected materials.
- Once the material candidates are shortlisted by wavelength transmittance, the more conventional design process follows
   this when further mechanical, thermal and chemical resistance properties are of interest.
- 102 Finally, the cost analysis needs to be taken into account when the material type providing the optimal solution is chosen.
- 103 Table 1 summarises some of the most related properties of a selection of practical optical materials. As expected, all of the
- 104 listed properties are functions of temperature, size and shape, exact composition, heat treatment, surface finish, and other 105 manufacturing processes. It is important to note that there are significant differences (10-15%) between the claimed values
- 106 by different manufacturers and textbooks.
- 107

# Table 1. Optical material properties

	Unit	Soda Lime Glass	Borosilicate	Quartz	Fused Silica	Sapphire	Magnesium Fluoride
General							
Chemical Formula, Composition	(weight %)	SiO2:74, Na2O:15, CaO:5, others	SiO <sub>2</sub> :80+, B <sub>2</sub> O <sub>3</sub> :7-13, Na <sub>2</sub> O, others	SiO <sub>2</sub> :99	SiO2:99	Al <sub>2</sub> O <sub>3</sub> :99	MgF <sub>2</sub> :99
Density	(g/cm³)	2.2-2.52	2.2-2.4	2.2	2.2	3.98	3.18
Optical							
Useful Transmission	(nm)	320-2300	325-2100	200-2400	180-2200	150-5000	110-7500
Refractive index (588 nm)	-	1.52	1.47	1.46	1.46	1.76	1.38
Mechanical <sup>1</sup>							
Young's Modulus	(GPa)	72	64	73	73	335	138
Tensile Strength	(MPa)	41	27-62	50	50	275 "	140
Hardness, Vickers	-	550	520-580	1000-1200	1000- 1200	1940	400
Poisson's ratio	-	0.23	0.21	0.17	0.17	0.25	0.27
Weibull variability of strength	-	6 ""	30 <sup>IV</sup>	8.82 <sup>v</sup>	10.2 <sup>VI</sup>	5	5
Weibull stress	(MPa)	129 💷	71 <sup>IV</sup>	115 <sup>v</sup>	180 <sup>VI</sup>	485	96
Thermal							
Softening Point	(°C)	1450	800-850	1730	1600	2300 VII	1255
Max. Continuous Operating Temperature	(°C)	260	280-350	950-1150	950-1100	1200	500
Thermal Conductivity at 300 K	(W/mK)	0.96	1.1-1.2	1.38	1.38	27.21	11.6
Coefficient of Expansion	(10 <sup>-6</sup> /K)	3.5-9	3.25-4	0.55	0.55	8.4	8.9

properties perpendicular to optical axis

materials are birefringent for exact refractive indexes see references

mechanical properties at room temperature

<sup>1</sup> mechanical and optical properties are dependent on fabrication method and surface finish; <sup>II</sup> fractural strength; <sup>III</sup> Kimble R-6; <sup>IV</sup> BK-7; <sup>V</sup> standard polish; <sup>VI</sup> "super polish"; <sup>VII</sup> melting point

108

Soda lime glass is the common glass type that can be found everywhere. It is mass-manufactured by floating the hot raw material on a bed of molten tin. It is the least expensive material of all, and being softer than other glasses, it is easy to make a complex part out of it. It is a hard material with good scratch resistance, but is significantly softer than other glasses or sapphire. It is not resistant to many chemicals, and its higher coefficient of expansion makes it sensitive to uneven temperature distribution [28-32].

Borosilicate glass is 2-3 times more expensive than soda lime glass but still considerably less expensive than fused quartz or silica. It has the same easy manufacturing properties as soda lime but usually has a lower thermal expansion coefficient, hence making it more resistant to thermal shock. Leaching can occur but it is more resistant to chemicals [28-31, 33, 34].

117 Fused quartz and silica have very similar properties as they have an almost identical composition. The main difference 118 between them is in the amount of contamination caused by the different manufacturing processes. Quartz is made from 119 melted and cleansed naturally occurring quartz sand with larger amount of contamination in the product, while fused silica 120 is a pure version of quartz synthesised from various gases. However, their mechanical and electrical properties are identical. 121 The only contrasting (and significant) advantage is that, silica has an excellent transmittance in the ultra violet (UV) region. 122 This property makes it unique among silicon oxides. A major advantage of quartz and silica, when compared to cheaper 123 glasses, is their increased stability. Their mechanical properties are significantly less sensitive to temperature changes than 124 borosilicate or float glasses. For instance, for a borosilicate, the linear thermal expansion at 500 °C increases its ambient 125 value a few hundred times; silica, however, faces an increase of about 40 times and then stays constant with further increase 126 of the temperature. This makes the evaluation of thermal stresses a lot easier when implementing quartz and silica. 127 Nevertheless, their excellent properties come at a price: the material cost is significantly higher than the aforementioned 128 glasses and their higher temperature resistance makes fabrication more complex. They have a reasonably good resistance 129 to chemicals but break down with some caustics, fluorinated acids and plasmas [28-31, 33-36].

Sapphire is a single crystal and a very versatile material. It is the second hardest material on Earth, which makes it best choice of material whenever wear and abrasion are the main constraints. Its high mechanical strength and modulus of elasticity provides good resistance against impacts. It is virtually impervious to all corrosive materials and its thermal stability outperforms all other optical materials. Yet, sapphire raw material is not significantly more expensive than fused silica. On the other hand, its extreme hardness and a high melting point make the manufacturing process challenging and costly. In conclusion, sapphire is not suitable for large windows and for complex shapes [37-43].

Magnesium fluoride is an excellent material choice for application in the UV bandwidth (the cheaper CaF2 has similar properties but with slightly reduced useful transmittance range). Larger size crystals can be grown, and it is possible to machine it with standard diamond tools as this material can be polished well. Thus, complex shapes and geometries can be achieved. It has a wide range of transmissivity but it is not as wear-resistant as the other materials, and its surface will degrade in a humid environment at elevated temperatures (over 500 °C) [33, 39, 44-46].

# 141 **3.2** Geometric design and mounting methods

142 There are a number of different ways to hold the optical element within an optical apparatus. A particular mounting method 143 can be selected considering the geometric constraints, the sealing requirements, position accuracy, the orientation of optical 144 axis, stress and the deformation caused by pressure difference, and birefringence. In this paper, sight optics is investigated 145 only; their mounts are less complex than lenses that need more degrees of freedom.



## 146 **3.2.1** Optical element kept in place by a guided clamp

147

148 Figure 3. Fixed volume combustion chamber with circular window that is positioned by a guided clamp [47].

Figure 3 shows the usual clamping method where the retainer is fitted and guided in the directon of the displacement of the window. The radial position of the retainer is fully defined by the contact forces. The advantage of this solution is the simple tensile load on fixing bolts, and simplified dismantling and re-assembly. Details of loaded bolted joints can be found in the literature [48]. The disadvantage here is that the larger the size in the direction of optical axis, the more complex its design and manufacturing turns out to be [49].

## 154 **3.2.2** Optical element kept in place by a free sitting clamp



156 Figure 4. Four-stroke optical engine, the rectangular window is sandwiched by the clamp and soft gaskets [6, 7].



#### 157

155

158 Figure 5. Special sodium chloride free sitting window for a high-temperature, high pressure difference, IR spectra [50]

The simplest design solution is illustrated in Figure 4 and Figure 5. The clamp is not guided but constrained by contact in one axis and constrained by friction along the other two axes; its position is defined by fixing bolts. Its advantages are: a simpler design, easier to manufacture, smaller in size along the optical axis, and that its position along the optical axis can easily be varied. Its disadvantage is that a greater amount of mechanical (bending) load on bolts is required; since the window can freely move, bringing the assembly together can also be problematic.

#### 164 **3.2.3** Adhesives

Fixing an optical element in a carrier frame using adhesives, as indicated in Figure 6 is a convenient solution for lower pressure and temperature environments. In both cases, the window sits against a shoulder which provides an accurate positioning. All mechanical loads rising from the pressure differential are taken by the adhesive. In the second case, the adhesive acts as a sealant and retainer; only, the stress is induced, but the pressure difference is taken by the shoulder on the frame cell. The main advantage of this solution is the modest space requirement. Its only disadvantage is that the performance of the assembly is proportionately dependent on the properties of the adhesive, which are usually limited.





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172 **3.2.4** Fitted inside the shell of the vessel



#### 173 174

Figure 7. Window integrated in the vessel body [53, 54].

The optical element can be fitted inside the housing, a typical application area is the deep submergance vehicles, Figure 7. There is no need for bolts as the major load-bearing element. Its advantage is that this setup can take large pressure differences, while the vessel geometry can also be made more simply. Its disadvantage is that the window can only be dismantled from the pressurised side, more complex window geometry required.

# 179 **3.3 Structural design and performance**

# 180 3.3.1 Allowable or design stress in the optical element, safety factor

The estimation of the allowable or design stress is among the most important and sometimes challenging tasks, especially at elevated temperatures [23-25, 55-57]. The data of mechanical properties can be found in the literature for the more common materials; however, there often is no consistency in the given values. It becomes even more difficult to find information when practical issues are being considered, such as the effects of temperature, humidity, manufacturing technology, surface finish, and loading rate. Pressure vessel codes provide suggestions for high strength alloys which can then be taken as a first guidance for optical materials. According to BS EN 13445-3 [56] and ASME Boiler and Pressure Vessel Code Section VIII [57], the design stress should be calculated as:

(EN) 
$$\sigma_{des} = \min\left(\frac{R_{p0,2/T}}{SF}; \frac{R_m/20}{SF}\right) = \min\left(\frac{R_{p0,2/T}}{1,5}; \frac{R_m/20}{2,4}\right)$$
 (1)

$$\sigma_{des} = \min\left(\frac{R_{p0,2/T}}{SF}; \frac{R_m/20}{SF}\right) = \min\left(\frac{R_{p0,2/T}}{1,5}; \frac{R_m/20}{2,14}\right)$$
(2)

188 where,  $\sigma_{des}$  is the allowable design stress; *SF* is the safety factor;  $R_{p0,2/t}$  is the 0,2% proof strength at T temperature;  $R_m$  is 189 the tensile strength at 20 °C [58]. As optical materials discussed in this work have brittle characteristics, it is only the safety 190 factors that are associated with the tensile strength that are applicable. It is suggested that the safety factor for optical 191 design should always exceed 2. The general value for a well-designed system is around 3, when failure is not expected to 192 cause major damage. When there is more uncertainty in the design, the usual and conservative safety factor value is 4. The 193 value can be as high as 5 for non-optimum or unplanned conditions (manufacturing or usage) or when failure can cause 194 significant damage [51, 59].

## 195 **3.3.2 Geometric and mechanical tolerances**

196 The tolerances on diameters and on the thickness of the centre and edges are comparable to general precision 197 manufacturing, typically h6 to h11. When the edge of the window is not fitted and/or it is not a sealing surface the size 198 tolerance is in the 0.1-0.01 mm range. The tolerance on the thickness of the optical access has importance for lenses but for 199 windows it is not crucial. Similarly, the usual parallelism requirements are on the fine side but are comparable but to the 200 ones used in pressure vessel manufacturing. However, the surface roughness values are in a couple of order less than the 201 typical values for precision manufacturing technologies for metals. Moreover, the quality of the finish is further described 202 by the scratch and dig number. A usual scratch/dig specification consists of two numbers e.g. 80/50. The first number 203 indicates the maximum size of cracks (scratches) on the surface of the optical element. The second number describes the 204 maximus size of round-shaped imperfections: digs and pits [60, 61].

205 Guidelines are given in Table 3 for selecting optical and mechanical properties for sight window applications.

## 206 3.3.3 Deflection and stress

Equations relating deflection to the applied pressure difference can be found in the literature for a number of shapes and support modes [62, 63]. In this work only the details of relations for the plane-parallel circular window shape are shown.

Table 2, Classical mechanics of plane-parallel circular elements



210  $K_w$  is a generalised constant suggested by textbooks [51, 52, 59]. In these works,  $K_w$  is choosen conservatively to cover a 211 wide range of optical materials. This conservative method was chosen in this study to make a suggestion for values of  $K_x$ . 212 In the rest of the equations, (3)-(10)  $\delta$  is the stress;  $\mu$  is the Poisson ratio;  $\Delta p$  is the pressure differential; r is the radius 213 which is half of the aperture or diameter  $D_0$ ; x is the defelction; t is the thickness of the optical element; E is Young's 214 modulus. If the the stress equations are rearranged and the safety factor, the diameter and design stress are inserted, then 215 the minimum required thickness of the optical element can be calculated.

$$t_{min} = \left(\frac{1}{2}D_o\right) \left[\frac{K_w SF_\delta \Delta p}{\delta_{des}}\right]^{1/2}$$
(11)

where,  $t_{min}$  is the minimum thickness of the circular optical element;  $D_0$  is the diameter of the aperture;  $SF_{\delta}$  is the safety factor;  $\Delta p$  is the applied pressure difference on the optical element;  $\sigma_{des}$  is the allowable design stress. Using Equations (7) and (8), the deflection can be calculated or the rearranged version with the maximum allowable deflection can be used to find the minimum required thickness.

219 find the minimum required thickness:

$$t_{min} = \left[\frac{SF_x K_x \Delta p D_0^4}{16 E x_{max}}\right]^{\frac{1}{3}}$$
(12)

where  $SF_x$  is the safety factor. In general, as  $SF_\delta$  is associated with complete breakdown and failure and  $SF_x$  has an effect on only the quality of the image produced by the optical element.  $SF_x$  can have a significantly lower value than the  $SF_\delta$ . Equation (12) provides results for a simple case of a mechanical load. When, there is a combined load from thermal and mechanical loads, the deflection needs to be calculated using Finite Element Analysis (FEA). The result of the simulation can be used to calculate the outer radius (R) of the window that can be turned into a divergent meniscus lens (assuming the same deflection on both sides of the window):

$$R = \frac{x^2 + D_0^2}{8x}$$
(13)

226 Then with the known thickness the lens power  $(P_{lens})$  can be calculated:

$$P_{lens} = (n-1)\frac{-t}{R^2 - Rt}$$
 (14)

The maximum deflection of a window is a function of allowable image distortion. In an optical system with lens and sensor,
the lens focusing error usually gives the tolerance in dioptres. It is hard to find tolerances published, but as a rule of thumb
some values are summarised in Table 3, [64-67].

230 Equations of stress, deflection and power calculation for rectangular, plan parallel windows can be found in [68].

#### 231 **3.3.4 Failure estimation by statistical tool**

232 It is a common practice to implement Weibull statistics to estimate the probability of failure ( $P_f$ ) when a given  $\sigma$  load is 233 applied on a brittle material.

$$P_f = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right]$$
(15)

where *m* is a constant describing the variability in strength; their values having been experimentally determined and published.  $\sigma_0$  is a stress level at which 63% of the samples fail, *m* is the so called Weibull modulus and indicates the scatter of fracture stress around  $\sigma_0$  [51, 69-72]. The acceptable values can differ significantly and they should be determined for each application individually. Some suggested examples: for a cheap easily replaceable cutting tool - 10<sup>-2</sup>; for an expensive part that upon failure can cause serious damage – 10<sup>-4</sup>; when personal injury is at risk – 10<sup>-6</sup>; when the outcome of a failure could be fatal then 10<sup>-8</sup>.

# 240 **3.4 Optical design and performance**

#### 241 3.4.1 Birefringence and maximum optical path difference (OPD)

It is usual for most practical optical materials to have two indices of refractions. Their refractive index is a function of the propagation-direction and polarisation of the incident electro-magnetic wave. Furthermore, it is a function of the mechanical stress in the medium. Optical substances having this property are called birefringent materials [73]. The level of birefringence is expressed as a difference in the optical path of two perpendicular states of the polarised wave. This inequality in distance is called the OPD and it is measured in nanometres. The OPD has been previously investigated for plane-parallel circular plates with a pressure differential applied on them; Sparks et al. [74] derived an approximate relation:

$$OPD = 8.89 \times 10^{-3} (n-1) \frac{\Delta p^2 D^6}{E^2 t^5}$$
 (16)

where, OPD is the optical path difference; n is the refractive index of the material;  $\Delta p$  is the pressure difference applied across the planes of the optical element; D is the aperture, the unsupported diameter of the optical element; E is Young's modulus of the medium; and t is the thickness of the window. This OPD caused by an applied stress called the stress birefringence. It is measured as OPD per unit travel path; its unit is nm/cm. The details of the maximum allowable tolerances on birefringence for some applications are given in ISO 10110-8 [61] and Kimmel and Parks [75]; a summary is presented in Table 1. Equation (16) can be rearranged to find the minimal required thickness:

$$t_{min\,|ODP\Delta p} = \sqrt[5]{8.89 \times 10^{-3} (n-1) \frac{\Delta p^2 D^6}{\text{OPD} \cdot E^2}}$$
(17)

254

#### Table 3. Sight window properties for different applications

Precision	Typical application	Maximum power of a deflected window (dioptre)	Maximum OPD per unit path length (nm/cm)	Parallelism or plane angle (degree)	Flatness (A is the characteristic wavelength)	Surface finish or roughness (nm)	Surface finish quality
Extreme	Polarisation and interference instrumentation, deep-space instrumentation	No data	2	No data	$\frac{\lambda}{20}$ or better	0.3	No data
High	Photolithography optics and astronomical telescopes	No data	5	0.001	$\frac{\lambda}{10}$	0.5	10/5
Good	Photographic and microscope optics, visual telescope	$10^{-2} - 10^{-6} *$	10	0.01-0.001	$\frac{\lambda}{2} - \frac{\lambda}{4}$	1	40/20- 20/10
Semi	Eyepieces, viewfinders, magnifying glasses	10 <sup>-1</sup>	20	0.1-0.01	λ	2	60/40
Commercial	Illumination optics, condenser lenses	No req.**	No req.	0.1	No req 2 λ	4	80/50

\*in general it can be said that, tolerance values in the order of  $10^{-6}$  or less are likely to be negligible when they are compared to the uncertainty in the focus adjustment of a lens system

\*\* No req.: No requirement

It is important to note that there are always some residual stresses in optical materials, depending on the quality of the manufacturing processes. More details relating the manufacturing process to stress birefringence can be found in the references.

## 258 **3.4.2** Factors limiting the maximum thickness

- The most obvious limiting factor is the available space the geometric constraints, which depends on the individual design. The different possible mounting methods and previous publications of solutions are introduced in other sections of this work.
- Transmittance change as a function of material thickness [27]. Significant decrease of the transmittance can only
   occur with large thicknesses, this is not a usual design constraint for high load applications.
- Temperature gradients can cause stress concentration in window materials. For heated or cooled designs this can limit the size of the geometry. Ceramics with larger thermal conductivity coefficients are less sensitive to thermal shock, [76, 77].
- 267 Price: manufacturing and material cost
- 268 As Figure 8 indicates typically there is a thickness range where the price is at its minimum. This is the most 269 commonly made size range( $t \leftrightarrow pt; p \sim 3 \dots 5$ ) that is mass produced with a variety of tolerances and finishes. 270 These are usually used in general optics and not adequate for high load applications. To the left from this region  $(t \leftrightarrow mt; m \sim 0.6 \dots 1.0)$  there is an increase in price where the manufacturing becomes more laborious. The 271 272 relatively thin geometry makes the window fragile and prone to deflection under the manufacturing loads. A 273 further sharp rise expected for thicknesses below mt where extra care is required to provide precision finish. For 274 example, during manufacturing there is a 50% breakage rate for 0.2mm borosilicate glass coverslips. On the right 275 hand side from the flat  $(pt \leftrightarrow nt; n \sim \text{few hundreds})$  the increase is driven by the cost of material. nt represents

- the maximum size that is achievable using the standard or already existing raw material production tooling. Larger
   geometries can only be made if tooling cost is covered.
- Figure 8 only introduces general trends in the price the actual values will differ from geographic region to region, material type, and quantity required.



Figure 8. Approximate cost of manufacturing of disc-shaped windows as function of thickness

# 282 **3.4.3 Other design considerations**

In this section further design considerations are listed and referenced. They are not of interest to this study, but they can
 be potentially important for other designs, for instance, in applications where the pressurised chamber is used with high
 accuracy polarisation or interference instruments or deep space applications.

- Compressive stress caused by sharp edges on the surface of an optical element [78, 79]
- 287 Effect of a temperature gradient on adhesive bonds [52, 80]
- The tensile stress in a brittle material due to a compressive load on its surface [52, 63, 81]
- Focus shift in thick parallel plane optical elements [82]
- Distortion caused by a temperature gradient [71, 83-85]

# 291 **4** Design of an optically accessible pressure chamber

The detailed geometry of the existing combustor is shown in Figure 9. An important feature of it is that the working chamber was constructed from sections. Utilising this property, the reactor's length could be varied to adjust the residence time of the reactants. Different length sections were available to build the reactor. When the reactor was assembled, the sections were sealed by polymer O-rings. As a result of the limited temperature resistance of the stainless steel structure and the high thermal load, the sections had to be individually water-cooled, as in Figure 9.



Figure 9. An isometric and a section view of existing chamber, computational result of in-cylinder temperature distribution shown in the section view. Conditions: diesel fuel; stoichiometric ratio; 6 bar in-reactor pressure.

300

313

# **301 4.1 The section**

# 302 4.1.1 Design requirements

It was required that the optical section withstand the maximum of 20 bar working pressure at the maximum possible 303 304 operating temperature and that its geometry would allow it to be connected it to the existing rig. In order to seal the reactor, 305 it was essential to maintain the temperature at an acceptable level in the O-ring grooves. The maximum continuous 306 operating temperature of the Viton O-rings (200 °C) was chosen as a limit on the surfaces that were in contact with the rings. The system could provide a maximum cooling flow rate of 10 litres per minute for the new optical section. The highest 307 308 allowed inflow cooling temperature was 70 °C. The estimation of the heat flux coming from the combustion to the section 309 was based on a number of test results where an in-chamber, single-point gas temperature measurement was taken. An 310 example of these results is shown in Figure 10. The results of computational work on combustion and in-chamber conditions 311 by Demosthenous and Crookes [10] were used as input boundary conditions for the analysis, Figure 9. It was also a 312 requirement that the windows could be easily changed to metal blanks for heating up or non-optical tests.



Figure 10. Typical test results of the existing combustion chamber; the time of ignition is indicated by the rapid increase in temperature and a pressure peak at around 10s after ignition

316It was an underlying requirement that the new apparatus would allow investigation with a Phantom 4.3 high speed camera,317a TSI Particle image velocimetry system (laser: dual 50 mJ/pulse, NewWave Gemini Nd:YAG; sensor: PowerView 4MP) and

318 a FLIR Titanium 560M infrared (IR) camera.

#### 319 4.1.2 Material choice

320 As shown in Figure 12, the resultant optical section is a complex shape, featuring fine finished surfaces for sealing purposes. It is thus that the material of the section body needed to have adequate strength to withstand the pressure load at high 321 322 temperatures. It also needed to be suitable for precision subtractive and additive manufacturing. Moreover, due to the 323 corrosive products forming inside the chamber and the constant presence of cooling water, the material was required to 324 have some corrosion-resistant properties. A detailed list of possible materials can be found in EN134453 [86]. The 325 aforementioned requirements suggested using an austenitic stainless steel grade. After considering the cost, the corrosion 326 resistance and manufacturability grade 304 (1.403) was chosen. It is easily available, with well-documented data on its 327 mechanical properties at elevated temperatures showing the tensile stress for the materials that were used to construct 328 the optical combustor, Figure 11.

The material selection procedure for the combustor body and for the optical element was based on general guidelines with practicality and availability in focus. However, in design tasks where material cost or other material attributes have higher significance more detailed material selection methods could be employed, as described in [87, 88].



332 333

Figure 11. Permissible design stresses of some selected materials as function of temperature [37, 56].

#### 334 4.1.3 Design of required geometry and validation

This structure of the overall design defined some underlying properties of the optical access. It was the obvious choice that

the optical access should be constructed on a section which has similar dimensions and had the same sealing method. Given

the nature of the laser-radiation-based measurement method, a three-access point – in a T-like configuration – was

338 required. A detailed review of optical measurements can be found in the books by Zhao [12, 13]; see details in Figure 12.

This three-access point design was satisfactory for the high speed and IR camera setup.





At its maximum performance the HPC burns approximately 4 g/s Diesel fuel; it can be seen that cooling is essential for the continuous operation of this reactor. Insufficient cooling would result in a rapid increase of temperature in the body and in the window (or blank). The excessive thermal load could lead to the quick failure of the polymer seals. Therefore, the geometry of the body with window seats had to provide enough surface area for the coolant and allow sufficient volume flow. Assuming the largest heat flux and inflow cooling temperature, a number of simulations were carried out to estimate the temperature distribution of solids and the coolant.

Full three-dimensional numerical model was implemented in SolidWorks Multiphysics modelling package using finite element method. Linear tetrahedral 4-nodes elements were used to discretise the solid components for the structural analysis whilst hexahedral cells were adopted for the fluid dynamics. The number of cells for the solid and fuild subdoamnis were 94710 and 62174 respectively, from which 66683 solid cells were in contact with the fluid.

For the purpose of structural analysis, a fixed constraint was applied on the conecting surfaces at one side and an axial evenly distribute load was added on the relevan surfaces other end of the section. Finally, a pressure load of 2 MPa was applied on the internal surfaces. The flow analysis was deifend by setting the temperatures, pressures and mass flow rates for the cooling flow and hot air inside at the inlet and outlet boundaries. The mesh and grid independence was investigated by means of software embeded tools where the mesh and grid were refined taking the notch effect, stress concentration and thermal singularities into account. Once the model was set up the coolant flow rate was varied and the temperatures were monitored, especially the temperature of the O-ring groove as shown in Figure 15 b.

The validation of the model was carried out experimentaly. The temperatures of the coolant entreing and leaving the section was measured and recorded. Moreover, the outside surface temperature of the metal blanks was monitored for validation purposes. It was found that the difference between the temperature values predicted by the numerical model and measurements was approximately 15%.

The O-ring groove temperature curve was plotted against the coolant flow rate, Figure 13. It was found that having at least 5.5 litres per minute coolant flow rate on the designed geometry could keep the O-ring groove temperature at an acceptable level. The 5.5 litres per minute minimum cooling flow requirement is below the 10 litres per minute maximum performance, therefore the given cooling system was found to be sufficient.



Cooling water volume flow (litres/minute)

367

378

Figure 13. The O-ring groove temperature as the function of the coolant volume flow (value was taken from the corner point of the hottest possible cross section, as indicated in Figure 15b).

370 The length of the section was chosen to be the same as that of the longest existing section. The thickness of the optical 371 section was based on the existing design. Polymer O-rings were used to seal between the sections, the design and 372 manufacturing were according to BS ISO 3601 [89]. With the given length, a circular window type was selected for easier 373 machining and the geometrically maximum possible diameter, 82 mm, was chosen to be evaluated. FEA was carried out to 374 determine the stress arising from thermal loads and applied pressure. As is indicated in Figure 14, the highest stresses are 375 in the O-ring grooves and in the openings. The grooves had high stresses on their contact surfaces because of the large axial 376 force pressing the sections together against the pressure. In the case of the openings the high stress rate can be explained 377 by the reduced material volume, i.e. reduced inertia [90].





379 The inner diameter of the optical section is 144 mm; the outer diameter is constrained by the support rods, as seen in Figure 380 9. As the radial space was limited, the best design solution would have been to place the optical element inside the shell – 381 but with the given casted base, this was not achievable. Therefore, the second best option - with an eye to optimising 382 storage space – was to have a free sitting clamp; this setup is shown in Figure 12. The detailed section-view shows the 383 window kept in place by the circular clamp. The clamps were fixed and positioned by 12 M6 socket-head bolts to the window 384 seats. The window seats were welded all around to the base chamber or reactor body; the beads sealed the seats and kept 385 them in place. The window to seat-sealing surfaces were precision-manufactured, as suggested in PD5550:2009 Table 3.8-386 3 [91]. With the aid of the measurements and computational results, further analysis was carried out to estimate the working 387 temperature of the section and optical elements. The temperature distribution of the cross-sections is shown in Figure 15.

# 388 4.2 The optical access

In this section the design process of a plan parallel circular optical element is introduced via a case study. Table 4 simplifies the procedure into four main steps showing all the properties that have effect on the design. In the following subsections a practical application of Table 4 is presented. The values for the variables and main functions/relations are cross-referenced from Section 3.

1, Determining design specifications:				
Collection of information that is required to carry out the design task.				
Instrumentational requirements				
	<ul> <li>EM range /Sensor responsivity (nm)</li> </ul>			
	<ul> <li>Transmissivity (%)</li> </ul>			
	<ul> <li>Window aperture (mm)</li> </ul>			
Nature of application, pre	cision			
	<ul> <li>Power from deflection (dioptre)</li> </ul>			
	•ODP (nm/cm)			
	<ul> <li>Parallelism (degree)</li> </ul>			
	<ul> <li>Flatness (fraction of characteristic wave length)</li> </ul>			
	•Surface finish (nm)			
	•Surface quality			
	<ul> <li>Properties of thermal and mechanical loads</li> </ul>			
	Chemical environment			
	•Safety factor			
Geometric requirements				
	•Constrains in all directions (mm)			
	• Window aperture (mm)			
	<ul> <li>Sealing surface area and shape</li> </ul>			
2, Material selection: This task can be carried out considering the following material prov	portion			
	•EM range (nm)			
	•Transmissivity (%)			
	Compatibility with environment			
	• Availability			
3. Thickness determination				
Calculation of the minimum thickness:				
This a usual undertaking where the minimum amount of material is without any failure. Numerical methods can be used to decrease the	determined that still ensures the window performing ne uncertainties in the result.			
	<ul> <li>Structural analysis, maximum stress (MPa)</li> </ul>			
	•ODP (nm/cm)			
	<ul> <li>Power from deflection (dioptre)</li> </ul>			
	Probability of failure			
Maximum thickness:	-			
The maximum window size is restricted by the followings.				
	<ul> <li>Constrains in all directions (mm)</li> </ul>			
	•Transmissivity (%)			
	Cost constrain			
4, Manufacturing details and instructions				
The tolerances and finish of an optical element depends on the application and precision required. The overall size is a				
result of the thickness determination and geometric constraints.				
	•Geometry: aperture, sealing area, diameter,			
	Parallelism (degree)			
	•Flatness (fraction of characteristic wave length)			
	• Surface finish (nm)			
	•Surface quality			
	- Junice quality			

394

# 395 4.2.1 Determining design specification

The main purpose of the window is to provide a transparent barrier between the combustion and instrumentation. An underlying requirement was to allow use of three different sensors (cameras) with differing spectral responses. Wavelengths of interest are particulate imaging velocimetry (around 532 nm), high speed camera (visible spectra) and IR camera (3 to 5 micron); the responsivity curves are shown in Figure 2. As the wavelength of the laser is in the visible range, there were two bands of electromagnetic radiation that needed to be considered – 380-985 and 2800-5200 nm. In these regions, the 401 minimum of 80% transmittance was required. The tolerance on the OPD had to be kept in the photographic range: 10 402 nm/cm. As the three sensors were robust and the measurements by them were not overly sensitive, the maximum allowed 403 lens power of the distorted window was 10<sup>-6</sup> dioptre. The maximum expected pressure difference on the optical element 404 was 20 bar. The required safety factor was required to be four for stresses arising from mechanical and thermal loads, with 405 a maximum probability failure of 10<sup>-4</sup>. The technological considerations and tolerances were chosen to fit laser and the 406 precision measurements requirements. The level of precision was selected to be good according to Table 3. Finally, the 407 operating temperature of the window had to stay under the maximum permitted level.

# 408 **4.2.2** Material selection: transmissivity and environmental requirements

409 The spectral requirement is shown in Figure 2 along with the transmittance curves. The ideal design solution was to select 410 only one material type to cover the required wavelength ranges. It can be seen that the two possible material types that 411 cover the needed large range of EM wavelengths are sapphire and magnesium fluoride. The thermal analysis of the optical 412 section indicated that the steel blanks and windows would need cooling to survive. The calculations and simulations were 413 carried out for both materials. It was found that MgF<sub>2</sub> can be a valid option for low-temperature and low-humidity 414 environments. Extra caution is required when a temperature gradient is applied on the MgF<sub>2</sub> material, as its high expansion 415 coefficient and middle-range conductivity combined with low strength makes it sensitive to thermal shock. It is also 416 suggested by manufacturers that MgF<sub>2</sub> can react with high temperature steam similar to the one that can be found in the 417 HPC as a combustion product. Therefore, sapphire was, instead, chosen as material for the windows as it combines good 418 transmittance in all the required wavelength bands as well as having good thermo-mechanical strength.



Figure 15. a) An isometric view of the flow of cooling water, its temperature change and the temperature distribution of a cross section of the reactor. b) Temperature distribution around the O-ring groove.

# 419 **4.2.3** Determination of required thickness and validation

420 The diameter of the window was determined by the maximum available space in the optical section. The maximum possible diameter was found to be 82 mm. Previous experience showed that a width of 9 mm minimum contact surface is required 421 422 to provide an adequate sealing performance. The 9 mm wide contact ring also ensured an acceptable level of compressive 423 stress in the window and provided large enough heat transfer surface for the metal blanks. This geometric design resulted 424 in a 64 mm aperture. The thickness of the window was estimated by using the equations and relations that were explained 425 earlier in this paper, and then validated by FEA. The minimum thickness was calculated for two requirements: maximum 426 allowable stress and OPD. Substituting values to Equations (11) and (17) the thickness value results were 5.88 and 0.07 mm 427 respectively.

$$t_{min\,|\delta} = \left(\frac{1}{2}D_o\right) \left[\frac{K_w \, SF_\delta \, \Delta p}{\delta_{des}}\right]^{1/2} = \left(\frac{1}{2}64\text{mm}\right) \left[\frac{0.75 \cdot 4 \cdot 2\text{MPa}}{178\text{MPa}}\right]^{1/2} = 5.88\text{mm}$$
(18)

$$t_{min|ODP\Delta p} = \sqrt[5]{8.89 \cdot 10^{-3} (n-1) \frac{\Delta p^2 D^6}{0 P D \cdot E^2}} = \sqrt[5]{8.89 \cdot 10^{-3} (1.76-1) \frac{(2MPa)^2 \cdot (64mm)^6}{10 \frac{nm}{cm} (345 GPa)^2}} = 0.07 \text{mm}$$
(19)

The results of the estimation indicated that, with the given loads and geometry, the required optical performance was easily achievable. Then, the window deflection was calculated using FEA for the highest thermal and mechanical loads. The stress and deflection results were substituted in Equations (13), (14) and (15) in order to check the design for failure probability and image distortion. Equations (13) and (14) combined together gives the power of a distorted window as function of refractive index, aperture and deflection.

 $P_{lens} = \frac{64(n-1)tx_{(t)}^2}{2}$ 

$$Hens = \frac{64(n-1)tx_{(t)}}{\left(D_0^2 + x_{(t)}^2\right)\left(D_0^2 + x_{(t)}(x_{(t)} - 8t)\right)}$$
(20)

433 The power as function of window thickness for the given geometry is shown in Figure 16.



434 435

Figure 16. Power of distortion vs window thickness

It was found that the limiting factors were the probability failure and deflection. Based, on the curve above and financial
consideration the thickness was chosen to be 10mm. Using FEA, the maximum stress was found to be 42.8MPa, with this
level of stress:

$$P_f = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] = 1 - \exp\left[-\left(\frac{42.8 \text{MPa}}{485 \text{MPa}}\right)^5\right] = 5.35 \cdot 10^{-6}$$
(21)

439 It is likely that there is a high inaccuracy in this result of probability failure. The value of  $\sigma_0$  is function of a number of 440 variables, one of the most important ones is temperature. In the equation above the value for  $\sigma_0$  corresponds to room 441 temperature, and its value expected to be show high sensitivity to changes in temperatures and therefore the probability 442 failure result can only be received as a guideline. However, no data available for  $\sigma_0$  in the literature for elevated 443 temperatures

#### 444 4.2.4 Results and detailed design



445 446

Figure 17. Side view: final sapphire sight window design, where  $\lambda = 532$  nm

447

Figure 17 shows the final and detailed manufacturing instructions of the sapphire window. The material selection, calculation of geometries and tolerances specified were carried out according to Table 4. The stainless section was manufactured in-house and three sapphire windows were purchased from a specialist company. These were installed in the

- 451 section to make the optically accessible combustor as shown in Figure 12. The HPC with the optical access has been fired
- 452 and fully tested successfully. The rig is capable to give insight to the high-pressure combustion process, providing optical
- 453 data at different EM wavelengths. Figure 18 indicates the results gained from the working optical section using different
- 454 instrumentation.



Figure 18. Results from the optical HPC: a) PIV vector field of the flowing air fuel mixture, laser measurement. b) IR radiation image recorded from a hydrocarbon flame. c) A still image from high speed video, visible spectra.

# 455 **5** Conclusion

This work has investigated the design of pressure vessels equipped with optical access. A significant number of data sources were surveyed to produce a comprehensive review of the most related optical, thermal and mechanical properties for some optical materials. The database was presented in a way that allows for a convenient and direct comparison. It was concluded that larger safety factor values are required for optical element design, typically ranging from two to five depending on operating conditions, manufacturing technology, risks and hazards and so on. The safety factor selection procedure and criteria was clearly described. The high values suggested is the result of the inconsistency found in available data sourced from suppliers and from the literature of the thermal and mechanical properties of optical materials.

The little design criteria that was available on the topic in the literature was presented, while also considering practical, mechanical and optical design considerations. The aspects of the design for optical performance were described in-depth, with additions to the already published equations and relations. The utilisation of this database allowed for the design process of a pressure chamber, with optical element under high mechanical and thermal load, to be demonstrated, where, the required experimental rig needed to support research activity for a range of optical instrumentation.

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472

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