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A Cone Calorimeter for Controlled-atmosphere Studies

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Many fires occur in ambient atmospheric conditions. To investigate certain types of fires, however, it is necessary to consider combustion where the oxidizer is not 21% oxygen/79% nitrogen. The Cone Calorimeter (ASTM E 1354, ISO DIS 5660) has recently become the tool of choice for studying the fire properties of products and materials. Its standard use involves burning specimens with room air being drawn in for combustion. To facilitate studying fires involving different atmospheres, a special version of the Cone Calorimeter was designed. This unit allows controlled combustion atmospheres to be created by the use of bottled or piped gases. To make such operation feasible, a large number of design details of the standard calorimeter had to be modified. This paper describes the background for these changes and provides an explanation of how the controlled-atmospheres unit is operated.

INTRODUCTION

In building fires, the composition of the inflow combustion air can range from 21% down to 0% oxygen. The remainder can include not just nitrogen but also various products of combustion from an already-burning fire. In the case of pre-flashover fires, however, the oxygen content is likely to be close to 21%. For post-flashover fires, the oxygen content can still be high near the floor but may be very low near the ceiling. Most fire tests are conducted using an air stream of 21% oxygen for the combustion air supply. In general, this has not caused problems in predicting the behavior of post-flashover room fires, as evidenced by the success of specific correlations[1] or of actual engineering prediction methods.[2] Nonetheless, the development of correlations and prediction methods is still in its infancy. There remains the entirely real possibility that when more refined methods come to be evolved, needs for additional fire property measurements could arise. Such needs might well encompass combustion measurements done in non-ambient atmospheres.

Over the last few years, the Cone Calorimeter has been adopted by numerous laboratories as the tool of choice for measuring various properties needed to assess the fire hazard of materials or products. The basic design and features of the Cone Calorimeter have been described in a series of publications.[3–5] It has been adopted by the American Society for Standards and Materials (ASTM) as E 1354[6] and has also been published by the International Organization for Standardization (ISO) as DIS 5660.[7] In addition to the basic instructions given in the two test standards, a user's guide is available[8] which gives detailed operating and troubleshooting instructions. The important fire properties measured in the Cone Calorimeter include heat release rate, total heat released, effective heat of combustion, mass loss rate, ignitability, and the evolution of smoke particulates and various gas species. The use of the Cone Calorimeter to measure these quantities has been presented in references 9 and 10.

We especially note that the design of the Cone Calorimeter did not envision limiting its use to any particular category of fires or fire scenarios—it was to be a general-purpose tool capable of representing various fire conditions. Thus, if concern arises that exploratory studies with non-ambient atmospheric compositions should be performed, the Cone Calorimeter becomes attractive for use in such studies, largely because extensive data under ambient atmosphere conditions are already available from it. The design of the standard Cone Calorimeter was based on using room air, at approximately 21% O_2, as the combustion oxidant, both because such a value is, in fact, representative of many fires and because it leads to a relatively simple implementation. At the very early development of the Cone Calorimeter, however, it was contemplated that at a later date it might be desirable to evolve a non-standard research unit, wherein studies with various atmospheres could be conducted. This paper describes the development of such a research instrument.

At this point, we emphasize that no implication should be drawn with regards to conducting of standard fire tests. We are most definitely *not* concluding in this paper that the existing ASTM or ISO standards should in any way be changed to expand the range of fire test atmospheres. At present, we do not know, in fact, what are the errors that are made by using ambient-atmospheres data to represent all fires and all fire regimes (although the success in predicting the real-scale heat release rates in studies, such as cited above, would not have been possible had such errors been major). To help answer this question, it is necessary to first assemble some substantive amount of test data under non-ambient oxygen conditions. It was the goal of this project to design and implement an experimental apparatus for doing such studies. A few initial experiments under non-ambient conditions, however, have already been performed. Data from this investigation are being presented separately;[11] here we only describe the design and performance of the apparatus.

DESIGN DETAILS

Main variables

The controlled-atmospheres Cone Calorimeter was designed to enable three main properties of the external fire to be varied independently: (1) irradiance, (2) the volume

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flow of the combustion air flow, and (3) combustion air composition. The irradiance in the standard unit can cover ranges from 0 to 110 $\mathrm{kW\,m^{-2}}$; this is unchanged in the controlled-atmospheres unit. The standard unit does have provisions for varying flows, but, because of the open design, the flow can be varied only over a limited range. The normal flow is 24 $\mathrm{l\,s^{-1}}$. The fan has an upper limit of about 32 $\mathrm{l\,s^{-1}}$, while the lower limit of approximately 12 $\mathrm{l\,s^{-1}}$ is determined by the requirement that the flow must be fast enough so that no combustion products would fail to be collected by the exhaust hood. The combustion air composition, as will be set out in detail, below, can be varied over a wide range of conditions, limited primarily by the source of gases available to the user. By controlling both the irradiance and the air composition, a closer representation of the burning of a fuel element in the real-scale fire can be made. There are still aspects of real-scale fires which may not readily be represented in a bench-scale test (for instance, effect of scale on flame temperatures, or effects of geometry on flame quenching). Nonetheless, they are felt to be of lesser importance.

We point out that this unit does not have any provisions to alter the *total pressure* of the combustion chamber. This is consistent with the representation of fires in buildings, where the total pressure is, invariably, very close to ambient. Such a unit may not represent all of the fire conditions encountered in, for example, submarines or spacecraft. While those conditions may also need to be studied, it is impractical to attempt doing that in a Cone Calorimeter. Experimental combustors can be built inside of hyperbaric chambers.[12] However, these types of combustors share essentially no components in common with combustors which are designed for ambient pressure operation. Thus, there should be no expectations of readily generalizing this particular apparatus to non-ambient pressure studies.

General layout

Figure 1 shows a general view of the controlled-atmospheres Cone Calorimeter, Fig. 2 shows a schematic view, taken from the side-rear, and Fig. 3 identifies the major components and the layout. While some of the changes between the standard unit and the controlled-atmospheres unit are minor, the number of changes is large and affects nearly all of the systems on the apparatus. We have estimated that our cost for constructing the controlled-atmospheres unit was more than double that for the standard unit.

Enclosed specimen combustion chamber

Instead of being located in the ambient laboratory atmosphere, the specimen area is enclosed by Pyrex panels. The choice of glass as the material for the enclosure was dictated by the desire to accumulate as little of the heat as possible within the specimen compartment, and also to be able to observe the burning specimen. There are two glass doors for operating purposes, one at the front and one on the right side. The back door functions as a blow-out panel, to relieve excessive pressures, should such occur.

The flow of gases in the enclosed Cone Calorimeter is fundamentally different from the standard unit. In the standard unit, gases move through the system because the exhaust blower pulls them through. In the enclosed unit, however, gases need to be supplied, under pressure, to the specimen combustion chamber. This does not, however, eliminate the need for an exhaust blower. To realize that forced exhaust remains necessary one must consider the conditions within the combustion chamber. The combustion chamber is intended not to leak gases either in or out under modest pressure differentials. For example, joints and connections in the metal-work, which are normally butt-jointed in the standard unit, were sealed with a silicone sealant. While a leak-free construction has been strived for, a truly hermetic seal could not be produced for various practical reasons. Thus, it becomes important in the operation to not create an excessive positive or negative differential pressure. If such an excessive pressure were produced (which could occur by mismatching the exhaust rate to the gas supply rate), leaks could become significant and measurement accuracy diminished. Thus, the combustion chamber is equipped with a differential-pressure manometer, indicating the relative pressure difference between the outside and the inside. It was found that satisfactory operation could be obtained by maintaining the pressure inside the combustion chamber at 5 Pa greater than the outside. By ensuring that this differential is positive, rather than negative, it is possible to use data-reduction routines which rely on gas inflow (and not exhaust outflow) measurements. Use of such an algorithm requires that the duct system have good sealing and that leaks not occur prior to the point where the oxygen measurement is made in the exhaust duct. It did not prove difficult in this apparatus to achieve such a sufficient flow-tightness.

The heater and the shutter assembly

The heater assembly is basically standard, except that a water-cooled shutter has been added. In operation, a desired O_2/N_2 (or other gases, as appropriate) ratio is pre-set. As soon as the door is opened to insert the specimen, however, this ratio is disturbed because of air coming in through the door. Therefore, unlike in the standard Cone Calorimeter, testing cannot be started immediately upon putting the specimen on the load cell. Because the cone heater does not heat up to a constant irradiance instantaneously, it is not possible to insert the sample under a cold heater and to begin a test by turning power on to the heater. It is, likewise, not possible to equilibrate the heater to the desired irradiance, then open the door, put in the specimen, and immediately commence the test. If this were done, the specimen would receive, at the start of the test, a gas mixture which included the air blown in through the open door. What is required, instead, is a system whereby the heater can be equilibrated, the specimen inserted, the door closed, and yet the test not started until the atmosphere has returned to its desired pre-set value. To achieve this, a cold plate (a brass water-cooled shutter) has been provided between the sample and the cone heater. This shutter consists of a brass plate approximately 135 × 135 × 7 mm thick which has been milled with channels to circulate cooling water. The plate slides on a set of ball bearing compound glides

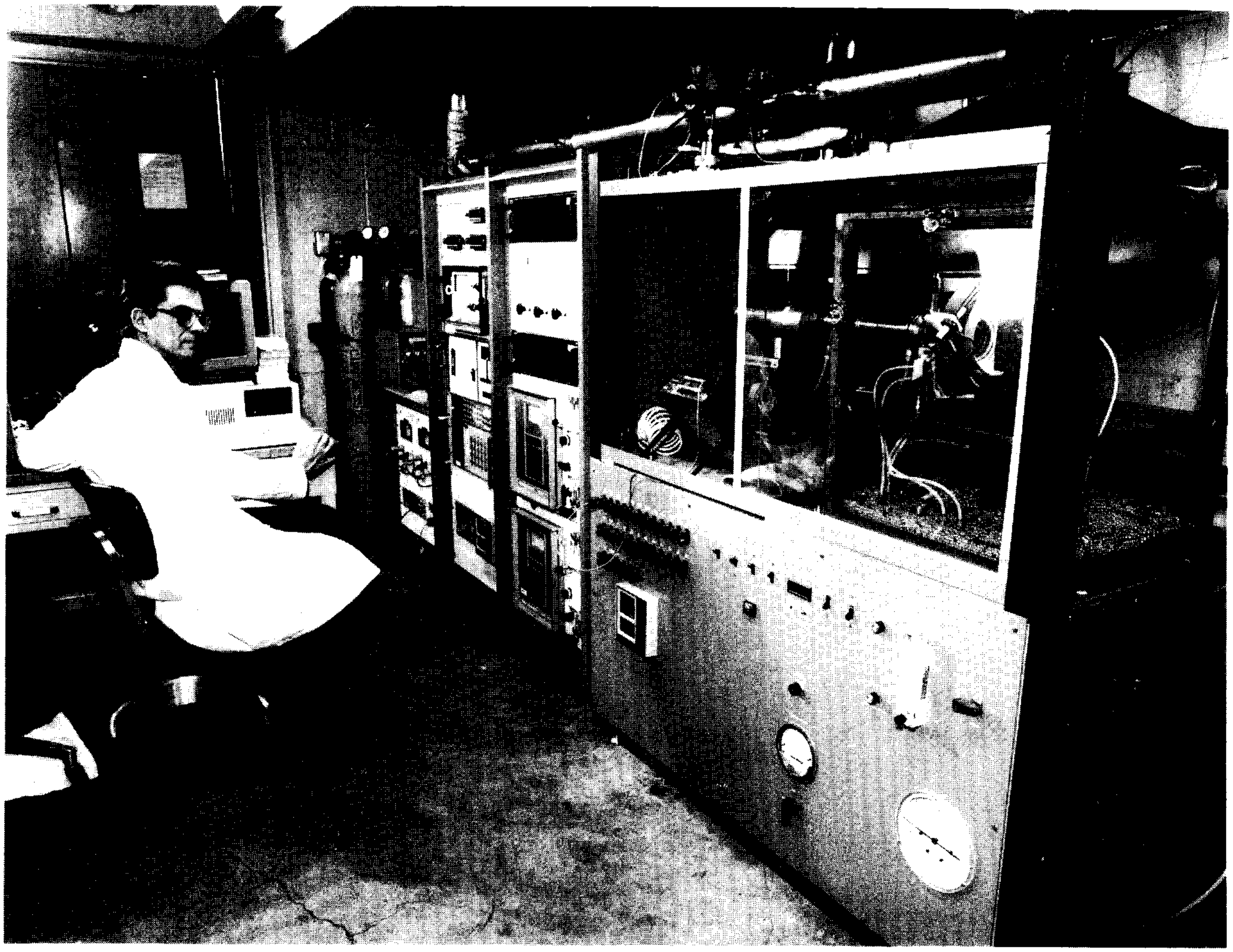

Figure 1. General view of apparatus.

which allow the plate to be moved in front of the heater to shield the sample.

The shutter mechanism has been designed with alternative means of actuation in the horizontal and vertical specimen orientations. In the horizontal orientation the shutter is moved by way of a 6.35 mm rod which extends through a gasketed hole in the left side of the enclosure. The operator grasps a knob on the end of this rod and, by pulling it away from the enclosure, slides the shutter out from under the heater, thus exposing the sample. By pushing this rod back in, the shutter will again block the sample from the heater. In the vertical orientation, the same shutter is used, but a different operating linkage is engaged. Because gravity is used to lower the shutter, the operator must open the enclosure and lift the shutter up in front of the heater and latch it on the rod each time before starting a new test. To slow the descent of the shutter and prevent it from slamming against its stop and disturbing the load cell a pneumatic 'dash pot' is attached to the shutter in this orientation.

Calibration heat flux meter and gas burner

The heat flux meter and the gas burner are identical to those in the standard unit. Their installation is somewhat different, however, since it is required that they be usable with the combustion chamber doors closed. Thus, gas, water, and electrical connections are made at special connector blocks located inside the chamber, slightly above the glass-bead bed.

Load cell

Despite the use of Pyrex glass side panels, it can still be expected that the specimen area temperatures will rise more than with the standard model. To allow for this, the load cell has been relocated to be below the bench-top. At the same time, the main height adjustment for the specimen was changed. In the enclosed Cone Calorimeter this is done by a height-adjustment device incorporated into the load cell carrier, rather than by moving the cone heater.

Spark ignitor

Because of the use of the Pyrex enclosure, a manual insertion of the spark plug holder into position was no longer appropriate. Instead, a pneumatically actuated

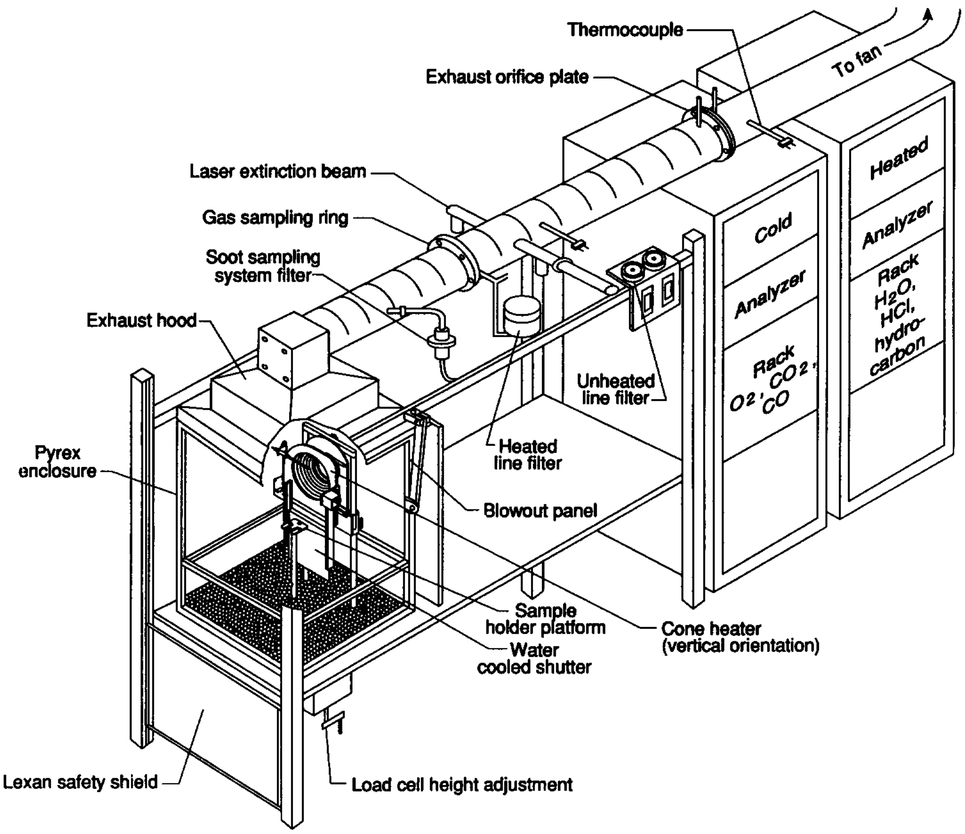

Figure 2. View from the rear.

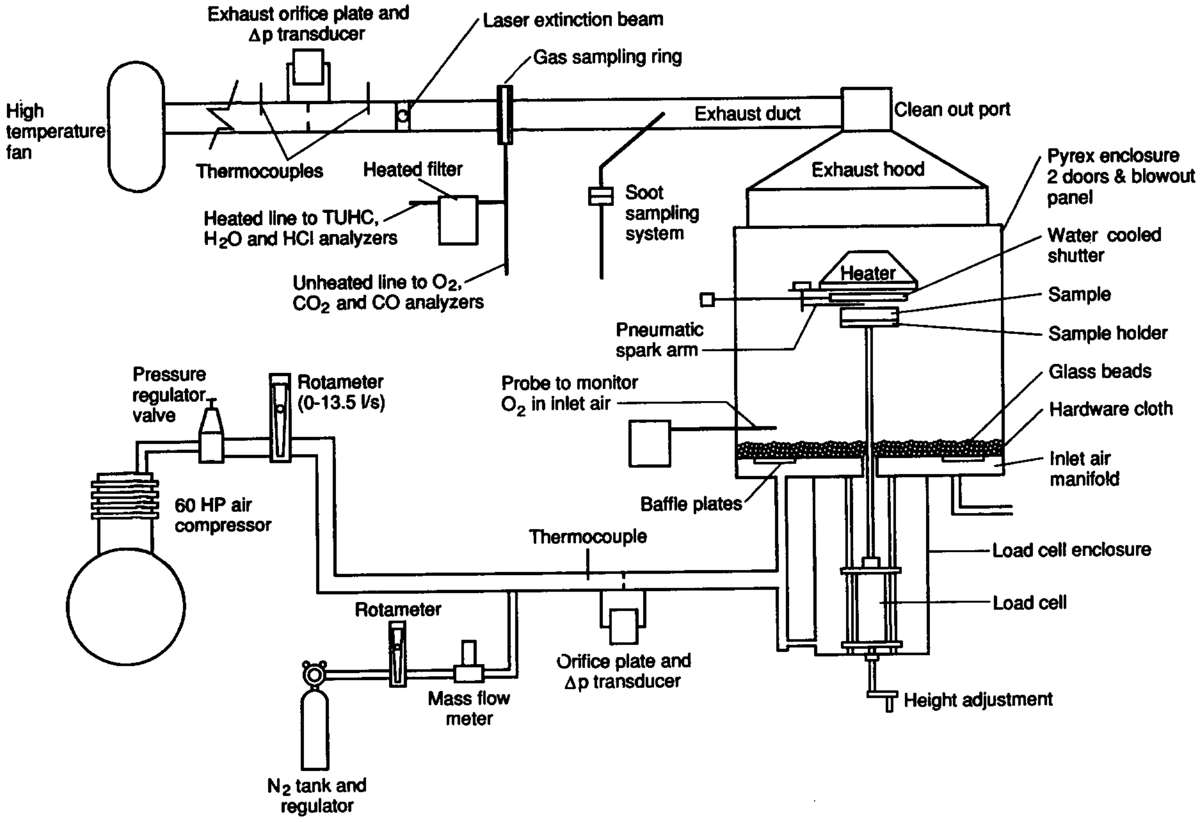

Figure 3. Functional diagram of apparatus.

spark plug holder was designed. The pneumatic circuit is, in turn, controlled by relays from the data computer. A different relay provides computer actuation of the spark high voltage. The two functions are not coupled, in order to leave freedom for the operator in case of specimen difficulties.

Supply and distribution system for the gases comprising the oxidant

The oxidant stream is introduced from the bottom of the chamber. Mixing of two or more gas streams is provided. The initial installation was equipped with: (1) dried and filtered compressed shop air and (2) nitrogen. The nitrogen comes from a liquid N_2 tank since the limited capacity of high-pressure gas cylinders would require their frequent replacement. The mass flow of the total mixed inflow stream is metered by an orifice plate, a ΔP transducer, and an associated thermocouple. Experiments also can, and have been, easily run whereby another gas, such as CO_2, is used, rather than N_2. For the case of CO_2, three cylinders with line heaters were used to provide adequate flow while avoiding freezing up of the valves.

The mixed air inflow stream is distributed into four inlet pipes going into the bottom of the specimen chamber. Above each inlet pipe is a baffle plate, intended to dissipate vertical momentum. The baffle plates support a wire mesh screen. Further velocity equilibration is done by passing the streams through a glass bead bed. The wire mesh screen serves to retain the glass beads in place. A fifth inlet pipe goes to the load cell enclosure. This purge is used to eliminate the need for a mechanical seal around the shaft used to support the sample. The balancing of flows going to the five inlet pipes is done with a gate valve located in each of the pipes. A new feature of the controlled-atmospheres Cone Calorimeter is the inclusion of a second oxygen meter. The probe for this meter can be alternatively selected for two locations: either slightly above the glass bead bed or in the inlet piping. In either case, it serves to monitor the oxygen content of the incoming gas stream.

In many experiments, the largest flow rate of inflow gas comes simply from a compressed air supply. This is provided by a 45 kW air compressor, located in a mechanical equipment room. This compressor supplies 860 kPa pressure air, which is reduced to near-atmospheric pressure by way of two single-stage regulators. This two-stage pressure breakdown helps maintain a constant flow. The second downstream regulator is used to set the desired air flow. Beyond this regulator is a particulate filter. The air then flows into two rotameters which gives the operator a visual check of the approximate inlet air flow. A pressure safety (blowout) valve at this point prevents damage to the rotameters should excess pressure build up in this line. The air line then goes to a 'T' which also brings in a second gas (N_2, CO_2, etc.). The air/gas mixture is then directed into a long straight pipe equipped with an orifice-plate flow meter located in the midlength for measuring the mass flow rate of the inflow. The gases then go into the manifold system, described above.

Exhaust system and ducting

Another substantial difference between the original unit and the controlled-atmospheres Cone Calorimeter is in the location of the exhaust fan. This is located right on the test bench in the original unit. In the controlled-atmospheres unit, provision was made for any desired future additions to duct instrumentation by locating the exhaust fan unit about 7 m downstream, near the building exhaust point. The instrumentation in the exhaust duct is basically similar to the original unit: an unheated sample line serving O_2, CO_2, and CO analyzers; a heated line serving total unburned hydrocarbon, H_2O, and HCl analyzers; laser smoke extinction meter; gravimetric soot sampling system; and exhaust duct mass flow rate measuring instrumentation.

Exhaust flows

The standard Cone Calorimeter procedure involves running at a flow of $24\,l\,s^{-1}$. With commonly available capacities of liquid nitrogen tanks, such a flow becomes difficult to achieve for the controlled-atmospheres unit. Instead, it has been found that a lower value is sufficient—this is in the vicinity of $10\,l\,s^{-1}$. This flow is rather low for the standard orifice size (1/2 of duct ID) used in the exhaust duct. Tests have shown that at this low flow, flow separation occurs and the original calibration no longer holds. For doing the controlled-atmosphere tests, data from the inflows are normally used instead of the outflows for data reduction purposes. To enable outflow rates to also be measured, however, an alternative exhaust orifice plate is inserted, one with a hole of only 3 cm. The minimum possible air flow is governed by the phenomenon of 'ponding' in the combustion chamber. If an excessively slow rate is selected, the exhaust products, instead of flowing through and out the chamber in smooth streamlines, will collect as if in an upside-down reservoir in the combustion chamber. Such a condition is undesirable for testing products, since the air being supplied to the combustion zone is no longer the inflow mixture, but, rather, has been vitiated by combustion products. Furthermore, additional reactions can occur as the gas stream submerges the heater. A condition of ponding can be readily avoided by visual observation of the combustion. The exact flow at which this will start to occur will depend on both the heater temperature and the heat release rate of the products. Generally, however, operation is successful down to $7\,l\,s^{-1}$.

Smoke meter

The laser photometer used for the standard Cone Calorimeter could not be used, since it depends for its operation on the assurance that the pressure inside the duct will always be negative with respect to the outside. This is invariably true with an open-air system, but may not be so with an enclosed one. If the relative adjustment of the supply inflow and the exhaust fan speed is such that the duct pressure at the photometer location is positive, then smoke will enter the optics of the photometer. Thus, for the controlled-atmospheres Cone Calorimeter, a forced air purge system was designed. A flow of $13\,cm^3\,s^{-1}$ to each side of the smoke meter was found adequate to prevent sooting up of the optics without artificially shortening the smoke path. Note that the smoke meter is located downstream of the analyzer sampling ring, so this introduction of room air does not affect the gas analysis.

SAFETY FEATURES

An enclosed combustion system, such as the one on the controlled-atmospheres Cone Calorimeter, can present a number of potential hazards which are not present when working with a standard, open-air Cone Calorimeter. Thus, as part of the design of this new unit, a thorough safety survey was made and a large number of special features were incorporated. Here we will briefly describe some of the major ones.

Piloted specimen ignition

If specimens were allowed to auto-ignite in this apparatus (as is quite properly possible in the standard Cone Calorimeter), a deflagration would occur within the combustion chamber. Several measures were taken, as explained below, to minimize the dangers associated with such an explosion. Nonetheless, prudent practice requires that no experiments be conducted without the use of the electric spark ignitor. Specifically, the spark ignitor must be 'on' at all times during the exposure of a specimen when the specimen is not flaming of its own accord.

Blow-out door panel

The combustion chamber is glazed on all four sides with Pyrex. The left side consists of a fixed panel and the front and right sides contain two doors, both used for normal operation and fitted with positive-sealing latches. The back panel is also hinged and can function as a door, but its primary function is to operate as a specially designed blow-out panel. The panel is mounted by a friction fit of a tang shape which protrudes from the frame and fits into a hole in neoprene gasketing located on the support frame. In the event of a pressure release, the pressure forces out the blow-out panel. Once such a release happens a hazardous condition could still occur if this panel were to collide into something and break or fall off the apparatus. Thus, it is equipped with a pair of connecting rods which limit the travel of the panel as well as preventing it from falling. This specially designed safety arrangement was proof-tested by creating an overpressure condition in the combustion chamber by high-pressure gas discharge. It was verified that the panel released when a pressure of approximately 0.7–1.0 kPa occurs. This pressure is well below the rupture range of the glazing, as well as below the yield strength of the latch mechanisms used to secure the two operating doors. The actual size of the blow-out panel used was 35 cm by 46 cm. This is consistent with a minimum recommendation[13] of 43 cm^2, based on the assumptions of breakage strength of glass being 20–30 kPa. Here we have taken the size of the chamber as 130 l.

Plastic protective shields

As a final line of defense against hazards due to explosion, sliding polycarbonate shields were installed on the front and right side doors/panels. These shields are slid out of the way during set-up, then pushed into place before starting a test. The left side panel also has a polycarbonate shield in front of it but is fixed in position. All three of these shields are separated from the enclosure by a 5 cm air gap.

Protection against an explosion from methane

The Cone Calorimeter is normally calibrated by the use of a calibration burner which is fed with a high-purity methane. If this burner were being used with the exhaust system turned off, an absence of pilot ignition spark, and the enclosure door closed, an explosion could result. To avoid this, an interlock system was installed which blocks the flow of methane to the calibration burner if the exhaust fan is not turned on.

Protection of flow meters

The flow meters and rotameters used to meter inflows of various gases could be potentially damaged, and possibly ruptured, if an over-pressure conditions developed in a feed line. Such a condition might result from a failure of a regulator or from operator error. To mitigate the consequences, the flow meters were protected by installing a blowout valve in front of each one. The valves were rated for 138 kPa release pressure, well below the 240 kPa maximum operating pressure rating for the flow meters.

Protection against a steam explosion

A steam explosion could occur inside the cold-plate shutter if the heater were turned on without water being circulated through the shutter. To avoid this, an interlock was installed which prevents the operator from turning on the heater while there is no cooling water flow. Further protection is obtained from the plastic shields, described above.

TEST OPERATION

The operation of the unit is generally similar to the standard unit, i.e. calibration of gas analyzers, etc. Thus, here we will only highlight those operating details which are different in the controlled-atmospheres unit. The heater is calibrated and the specimens prepared in the standard manner. The heater is first turned on and equilibrated to the correct temperature and flux. The desired inflow rates and ratios of O_2, N_2, or other gases are initially established by use of flow meters associated with each particular gas stream. An actual, precise oxygen concentration is monitored for the incoming air stream on-line with the data computer. Exhaust fan flow is set to obtain a very slight positive chamber pressure of about 5 Pa. The shutter is placed in front of the heater (by pushing in the operating rod for horizontal orientation testing; or by raising up and latching the shutter for vertical orientation testing). The combustion chamber door is opened and the specimen is placed in on the load cell platform. The door is closed and the operator then monitors the inlet and exhaust oxygen meters to be certain they both indicate the desired oxygen value before starting the test.

To start the test, the shutter is quickly removed by pulling on its control rod, which extends to the outside of the enclosure through a gasketed hole. This causes the shutter either to drop down by gravity (when the cone is in the vertical orientation) or to be slid back and away from the cone heater (when the cone is in the horizontal orientation). With the shutter removed, the heater radiation now impinges on the sample and the test has started. We note that the shutter only shields the sample from direct heater radiation. With prolonged heater operation, however, the combustion chamber tends to become warmer than the ambient air, so prolonged delays between inserting the sample and actually starting the test should be avoided to prevent a rise in the specimen's temperature before the start of the test.

With the shutter retracted, function keys on the data-collection computer are used to rotate into place the pneumatically actuated spark arm over the sample and to apply high voltage to the spark ignitor. The computer is then activated to take data scans of the various connected instruments, very similar to the standard Cone Calorimeter operation.

CLOSING COMMENTS

The unit installed at NIST has only been implemented for atmospheres containing 21% or less O_2. To use mixtures where $O_2 > 21\%$ requires installing systems for oxygen delivery. This is not difficult to do, but does require an additional area of anti-explosion protections, not discussed in the present paper. Because of the importance of elevated-oxygen combustion in certain aerospace applications, at least two aerospace agencies are currently constructing equipment with precisely this capability.

The paper describing the initial NST findings[11] on CO and smoke production from vitiated combustion suggests that the effect is not of major importance in determining fire hazard conditions (the study also describes under what conditions limiting available oxygen *can* cause significant hazard increases—the findings of that study should be consulted for further details). Thus, we do not expect a Cone Calorimeter with the features described here to be needed for engineering hazard assessment work. The unit, however, readily permits direct comparison of materials combustion, versus non-oxidative pyrolysis, which can be conducted by feeding 100% N_2 as the supply gas. This can be of significant advantage in the course of developing improved polymers. Thus, certain polymer producers are already installing this capability.

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