# IGNITION OF BULK SOLID MATERIALS BY A LOCALISED HOTSPOT

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Thermal decomposition of combustible bulk powders poses a risk in the process and allied industries in operations, storage and transport. Testing for storage and transport is well developed in establishing maximum possible pack sizes and safe ambient temperatures. However, current UN and EU tests are concerned with the maximum acceptable temperature for the whole bulk of the material, and do not consider the propagation from a hotspot (UN Manual of Tests and Criteria 2003, and BS EN 15188 2007).

This paper details the development of a small scale test method which has subsequently been used to determine the ignition and subsequent combustion/decomposition characteristics for various bulk solid materials initiated by a localised heat source. The results presented complement work undertaken by Brindley et al. (2001) to predict the ignition of weakly reactive solids by nearby heating sources via computational methods, and will also be used to assess the suitability of the small scale screening test; to be used as part of the thermal hazard assessment process to detect materials susceptible to this type of behaviour.

## **INTRODUCTION**

Testing for storage and transport is well developed in establishing maximum possible pack sizes and safe ambient temperatures. In particular the United Nations (UN) transport of dangerous goods regulations classify materials and determine acceptable packaging sizes. The materials of particular relevance here are those classified under the headings of 4.1 self reactive and 4.2 self heating substances. The UN tests however are concerned with the maximum acceptable temperature for the whole bulk of the material and do not consider the propagation from a hotspot.

Material currently tested is screened by a variety of tests, and this includes Differential Scanning Calorimetry (DSC) which can give an idea of the magnitude of possible exotherms, and their onset temperature. It should be noted however that DSC can miss some material behaviour which shows up in different tests, this can include reactions which are oxygen dependent, or endothermic decompositions which yield flammable gases which do not ignite in the DSC but do in different test equipment.

The UN 4.1 classification includes a raft of tests from the initial screen using Self-Accelerating Decomposition Temperature (SADT) and DSC to a series of additional tests including, Gap, Bonfire, Time Pressure, Koenen, American Pressure Vessel, Deflagration Burning and Lead Block test. Basket testing in ovens is used for UN 4.2 classification, testing at specified sizes and temperatures, looking for a sub or critical temperature (exotherm  $>60^{\circ}$ C) under the specified condition.

There are no defined tests for hotspot ignitions. Heated ball bearings have been used in some cases but these serve largely as a demonstration and communication tool, not as a robust test. These tests are largely unsatisfactory due to the difficulty of controlling temperature and the slightly arbitrary nature. This leads to confidence in positive results but not in negative results.

# HOTSPOTS

To understand the hotspot concept in process safety it is important to be able to identify hotspots in terms of what they are and where and how they are formed. Then as with other ignition sources they can be characterised using a number of different parameters, the four most critical being: temperature, total heat energy, size and duration.

Often in literature hotspots are categorised by the mechanism via which they are formed, e.g. friction, grinding, mechanical impact etc. In an intuitive attempt to identify them specifically by type they appear to fall into three distinct categories:

- Hot surfaces
- Hot fragments (sparks) most commonly metal
- Hot processed materials (powders)

A hot body or surface is produced via one of two methods. The first is by surface interaction between two bodies whereby mechanical energy is converted into heat typically through rubbing, grinding or impact. This is most likely to occur from wall blade contact or bearing failure in equipment with rotating parts such as screw feeders, pressure filters, fans, mills, mixers, centrifuges and dryers. It is often the result of misalignment or mechanical failure. A distinct type of hot body is a foreign object, commonly referred to as debris or tramp material, e.g. a nut or bolt or discarded tool. This is usually a problem in equipment with small clearances where the foreign object gets trapped between two surfaces one of which is moving, e.g. in mills and screw feeders. The tramp material may stay trapped and in effect behave like a hot body with an almost infinite amount of heat energy, or it may be dislodged and work its way through the process leading to a problem in a downstream vessel/item.

The second method is via the transfer of heat in process equipment leading to hot external (e.g. pipe) and internal surfaces (e.g. dryer wall). In the case of piping and dryers this can occur during routine operation as hot liquids and solids contact the walls, but for external casings on motors and pumps it is more likely as a consequence of electrical or mechanical failure.

**Small hot fragments** are produced when two bodies come into contact for a short time period resulting in a shower of hot fragments (sparks), also a hot body/surface is simultaneously produced. It is unlikely only a discreet fragment will be formed. This type of hotspot is most likely produced as the result of a short duration, high energy impact in equipment with fast rotating metal parts such as a motor, fan or high shear mixer, again most likely as the result of mechanical failure.

When **bulk powders** are processed at elevated temperatures or subjected to high energy impacts in mechanical equipment hotspots can be formed. They are commonly referred to as smouldering nests and are clumps of material undergoing combustion (typically oxidative). Often they are formed on the walls of dryers or in mills, mixers, extruders, and screw feeders. In a dryer they may reside on the wall for a long time period ultimately resulting in a combustion event within the equipment itself, but more commonly they become dislodged and are fed into bulk material further down the process, or are extracted into dust collection systems such as cyclones and filters. Typically they reside in bulk powder during storage or transport (in big bags or large containers, even silos), and because of their relatively small size they are extremely difficult to detect. They can oxidise very slowly for long periods resulting in full flaming combustion if sufficient oxygen is present.

# CHARACTERISTICS

#### TEMPERATURE

Hotspot temperatures vary significantly and depend very much on the type formed. Hot fragments exhibit very high maximum temperatures, typically well in excess of 1500°C (Pederson and Eckhoff 1987). Hot surfaces produced as a consequence of metal metal interactions can produce temperatures in excess of 1000°C (Hawksworth et al. 2005), but more typically in the range 500–1000°C (ISSA 1998). Hot surfaces produced as a consequence of over-heating electrical equipment are likely to be lower-T classification covers the range 85–450°C, with the majority of equipment rated T3 (maximum surface temperature 200°C). Typical temperatures for processed material undergoing a smouldering combustion are likely to be in the region 250–500°C (Krause and Schmidt 2000).

## HEAT ENERGY

The total heat energy output will again be dependent on the type of hotspot formed. In processes where the hotspot is

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generated by prolonged surface interaction, e.g. a stressed bearing, a significant amount of heat energy will be released. Power outputs from motors driving items of mechanical equipment will be in the order of kilowatts (typically 5-50) and the process of heat generation is often extended. So although only a fraction of the total power available will be dissipated in the hotspot, it is still likely to result in an almost infinite amount of energy available. In processes where the hotspot is generated by a short impact, a finite amount of energy will be available, although heat transfer rates will be high. In comparison, heat transfer rates for smouldering process material will be low but are likely to continue for prolonged periods resulting in a relatively high total heat energy release.

## SIZE

Fragments are small, typically 0.1 mm in diameter (Babrauskas 2006). Hot surfaces and bodies are wide ranging in size, a typical roller bearing is about 2–4 cm in diameter. A hot surface produced as the result of a mechanical impact will be small, whereas hot process equipment surfaces such as external motor casings or the internal walls of a dryer can be very large. Hotspots formed by hot process material also vary and they are dynamic too, obviously increasing in size as the smouldering front slowly propagates.

#### DURATION

The duration of the hotspot will vary, again depending on the type produced. Hot fragments and sparks will exist for only a short period whereas hot surfaces and in particular hot process material will persist for much longer often remaining undetected for weeks (Krause and Schmidt 1997).

# **EXPERIMENTAL ARRANGEMENT**

The basic experimental arrangement is detailed in Figure 1. It consisted of a constant power supply to an electrical heater (spherical hotspot). The hotspot was situated inside a cubic sample container with temperature measurement afforded by 5 thermocouples, 4 in the sample and one on the surface of the hotspot. The thermocouples and hotspot were held firmly within a block constructed from heat resistant material and the whole assembly was supported by a rigid steel frame.

The experimental arrangement was designed to replicate a scenario such as failure within a mechanical piece of equipment, e.g. bearing failure prior to collapse, with a spherical hotspot producing a constant, almost infinite amount of heat energy. As a result power was chosen as the sole experimental control parameter, and it was a relatively straightforward experimental control system to operate.

# POWER SUPPLY

The constant power control unit formed part of the power control circuit. It was essentially a power supply with a feedback amplifier to control the current and voltage



Figure 1. Experimental schematic

applied to the electrical heater. The control circuit was completed by an electronic watt-meter used to monitor the power of the heater, which was connected to the feedback amplifier. This control circuit allowed the power output from the heater to be controlled at a constant value throughout the duration of a test.

# ELECTRICAL HEATER (HOTSPOT)

The hotspot was simply a piece of wound resistance wire (nichrome) encased in a mouldable ceramic material to produce a spherical hotspot, see Figure 2. The ends of the resistance wire were welded to small lengths of stainless steel rod to provide stability and a method for connecting the hotspot to the power supply.

Nichrome wire is used to produce electrical heaters for similar small scale test work. It is the prescribed heater of choice for both the United Nations (UN) and European Community (EC) tests for evaluating oxidising properties in liquids (test O.2 and A.21 respectively). Also, it is



**Figure 2.** Hotspot design – moulded ceramic sphere on the left, brass sphere on the right

easy to manipulate, readily available and is relatively inexpensive.

After a number of trials using different thicknesses, 23 Standard Wire Gauge (SWG) was chosen for routine use. It contained enough physical strength to prevent premature burn out but was thin enough to be wound into coils for producing relatively small hotspots.

Once the coil was prepared it was necessary to encase it in a media capable of producing a spherical source. This shape was required to fit the specific process scenario (bearing failure) and the theoretical model of interest (Brindley et al. 2001). Also, a smooth spherical surface provided intimate contact between the test material and the surface of the hotspot. Nelson (1995), reports for purely hand wound heaters the coil tended to divide the powder and form cavities which prevented ignition.

A second generation hotspot was designed in which the resistance wire coil was embedded in a cavity inside a metal ball (brass). The cavity was filled with magnesium oxide to electrically insulate the coil from the brass ball.

#### THERMOCOUPLES

A mineral insulated Inconel 600 sheathed type K thermocouple with a swaged end was used in the experimental study. It was chosen as it offers many advantages: operable over a wide temperature range ( $0-1100^{\circ}$ C), fast response, flexible, rigid, suitable for arduous operating conditions and easily calibrated.

A bespoke thermocouple was manufactured at little additional cost. It consisted of a 1 mm diameter sheath that was approximately 500 mm long, incorporating a swaged end which was 0.6 mm in diameter. A small diameter sheath limited thermal conduction effects from the test material via the metal body but provided enough rigidity to ensure positional accuracy was maintained within the container. The tip length was 34 mm for thermocouples used in the 50 mm test material containers and 57 mm for those used in the 100 mm containers. These lengths were specifically chosen so that the thermocouples would sit in the central horizontal axis of the test material container.

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Three types of tip were available: insulated, ground (to earth) and exposed. The insulated tip provides a thermocouple junction that is well insulated from the metal sheath, whereas the junction in the grounded tip is physically bonded to it. In both of these designs the tip is only a 'few' millimetres from the end of the sheath. However, the junction in the exposed tip slightly protrudes from the end of the sheath. Ideally an exposed tip would have been most suitable particularly for temperature measurements on the surface of the hotspot, where the junction would have intimate contact with the surface to be measured. However, exposed tips can only be reliably operated to a maximum of 300°C and they are not robust so are totally unsuited to experiments of the type to be performed. The grounded tip offers the fastest response time but experience has shown that they are not nearly as robust as the insulated type. Therefore, an insulated tip was chosen as the most suitable option. The response time was acceptable, it copes well with test temperatures well in excess of 300 °C, and each thermocouple could be used successfully to perform a large number of experiments.

#### THERMOCOUPLE POSITIONING

The temperature gradient produced in a bulk powder by a localised hotspot is steep. Therefore, to have full confidence in the experimental data for this type of test it is extremely important that the position of the hotspot and thermocouples are known. Also, for accurate data interpretation it is important to replicate the positioning of the thermocouples from one experiment to the next. For these reasons a holding device (block) was designed, manufactured and utilised to hold the hotspot and thermocouples firmly in position. When fastened in the block the necessary rigidity and positional accuracy for the individual thermocouples and hotspot was provided. In trials, any significant disturbance to the block or surrounding area (e.g. test material container, fume cupboard base) did not affect the positioning in any way.

The holding block was constructed from Sidanyo<sup>®</sup>. This is a relatively new material that is finding numerous industrial applications, mainly as an asbestos replacement. It was chosen as it possesses excellent heat resistance properties, is easily fabricated and is reasonably inexpensive to purchase. Hollow metal inserts were incorporated within the block into which the thermocouples and hotspot were inserted. Once inserted, grub screws were used to clamp them tightly in place. A similar arrangement was used to hold the test material container in position.

#### TEST MATERIAL CONTAINERS

The test material containers used in the majority of the tests were stainless steel wire mesh baskets. This type of sample container is commonly referred to as a Bowes Cameron Cage and is used widely in industrial laboratories for performing small scale thermal stability tests. The simple design consists of a fine wire mesh cubic inner ( $\sim$ 50 µm) which sits snugly inside a more rigid cubic outer (mesh size  $\sim$ 200 µm). The outer cube is simply a means to

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provide physical strength for the inner container. The wire mesh arrangement allows free diffusion of air in and around the sample. This was important as at least one of the materials was likely to undergo an oxidative combustion. This type of container is available in 4 standard sizes of side length, 25, 50, 100 and 200 mm.

# SAMPLE DETAILS

The four samples chosen for the detailed hotspot studies were wood flour, hydroxypropylmethylcellulose, activated carbon (Norit), and an agrochemical formulation.

Cellulosic materials and carbon were chosen as they are representative of the types of materials handled routinely in the chemical and food processing industries, and have been involved in reported incidents. Also, the experimental hotspot studies previously published have predominately used materials of this type enabling some comparative analysis (Krause 1997 & 2000, Nelson 1995, Rogers 2006). It was also important to assess a material similar to that likely to be tested on a routine basis in the Syngenta Process Safety Laboratory. Therefore, the final sample chosen for detailed analysis was a typical agrochemical formulation.

## RESULTS

An extensive series of tests was performed in cubic containers of side 50 mm to investigate the no ignition/ignition boundary for wood flour using hotspots covering a range of diameters. At the same time, in order to establish the experimental repeatability a number of different hotspots were used with the same nominal diameter, i.e. for the 6 mm diameter heat source a no ignition/ignition boundary was determined for seven individual hotspots, nine individual 9 mm hotspots were used, six 14 mm, four 16 mm and one 20 mm hotspot. For an individual hotspot the lowest measured power producing an ignition is referred to as the critical power; the data is presented in Figure 3.

A similar series of critical determinations to that detailed above was also performed for the other materials. The results presented in Table 1 show the lowest measured critical power values obtained. Obviously, the values for wood flour are those marked by the crosses in Figure 3. The data sets for the other materials were not as extensive as for wood flour, 2 hotspots of each size were used with carbon, and 1 hotspot of each size for the other 2 materials.

#### TEMPERATURE

It was possible to establish no ignition/ignition temperature values in every test as the hotspot surface temperature was measured each time.

In tests where an ignition was not initiated a steady state temperature profile was established and the hotspot surface temperature was easily interpreted, see Figure 4. In tests where an ignition occurred a dynamic temperature profile was obtained as the sample exhibited exothermic activity, see Figure 5. To establish the hotspot surface temperature at the point of ignition a subjective 'steady state' baseline was added to the experimental data and the



Figure 3. Critical power determined in wood flour

point of deviation from the baseline was noted as the ignition temperature.

The range of no ignition temperatures for the wood flour experiments is shown in Figure 6. For a given diameter it is the highest no ignition temperature for each individual hotspot that is plotted. In the vast majority of cases this value was measured in the sub critical test, the sub critical test for an individual hotspot being where the highest measured power resulted in no ignition. The lowest critical ignition temperature determined for each set of hotspots is also plotted.

The no ignition temperature values were also examined for the other test materials and the lowest are shown in Table 2. Again, in the vast majority of cases the quoted no ignition value was that measured in the sub critical test.

#### SAMPLE VOLUME

A series of tests was performed using a single 16 mm hotspot to determine whether sample volume influences ignition. The results are presented in Table 3.

# HOTSPOT TEMPERATURE PROFILE IN AN INERT MATERIAL

Prior to any ignition tests a temperature profile was established for each hotspot by performing a series of tests in an inert material (Kieselguhr). The data obtained from the tests conducted at 3W is detailed in Table 4.

# DISCUSSION

# EXPERIMENTAL REPEATABILITY

The initial results obtained in the tests detailed in Figure 3 show the sub critical/critical power boundary was not sharply defined, as different hotspots with the same diameter produced a larger than expected range for critical power. Examination of the no ignition temperature ranges detailed in Figure 6 also shows a larger than anticipated spread. However, analysis of the respective data sets by determining a coefficient of variation for each hotspot size shows the spread of data across the range of hotspot sizes to be slightly better for the no ignition temperature measurements.

Initial concern regarding the non sharply defined critical region focussed on the experimental set-up and in

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	Critical power/(W)						
Hotspot diameter/(mm)	Wood flour	Carbon (Norit)	Hydroxy	Agrochemical formulation			
6	1.80	2.48	2.68	8.50			
9	2.50	3.04	5.01	8.34			
14	2.88	3.72	5.25	9.99			
16	3.72	4.00	6.61	10.29			
20	4.24	5.62	7.00	11.70			



Figure 4. Typical experimental trace for a no ignition test in carbon

particular the construction of the hotspots. This lead to the design and production of a perfectly spherical, altogether more concisely constructed 16 mm hotspot (brass ball). However, the data presented in Figure 3 shows the critical power range for the 16 mm hotspot is only slightly tighter than for the 9 and 14 mm ceramic hotspots, and no better than the 6 mm.

The packing density of the materials was examined but only slight variations in bulk density from test to test were noted. Furthermore, some worst case tests performed using extreme bulk densities produced little change in the critical measurement.

Finally, external measurements were taken to ensure the power provided by the hotspot was similar to that

being measured by the data logging system, excellent agreement was obtained in all cases.

#### CRITICAL POWER

The data presented in Table 1 shows all the materials tested exhibited sub critical/critical behaviour, i.e. for each material there exists a value for power which if exceeded results in the initiation of an exothermic combustion/ decomposition, and if not exceeded results in the establishment of a steady state temperature profile within the material. Although the critical boundary was not clearly defined for each of the hotspot sizes in wood flour, see Figure 3, the ignition/no ignition regions were clearly



Figure 5. Typical experimental trace for an ignition test in wood flour



Figure 6. Range of no ignition temperature and lowest critical ignition temperature determined in wood flour

established. The most likely cause for the poorly defined critical power region is the nature of the smouldering combustions being initiated. Small changes at the surface of the hotspot are likely to have significant effects due to the low reaction rates of oxidative smouldering materials previously reported (Rogers 2006). This is a fundamental problem with powder testing of this type. It is difficult to obtain repeatable, intimate surface contact between powder and hotspot from one experiment to the next due to the inhomogeneous nature of powdered materials.

For a given size of hotspot the critical power determined is different for all of the materials studied, and a consistent pattern was observed. The wood flour sample required the least power to initiate combustion/decomposition for all the hotspot sizes, in carbon the power was always slightly higher, and the values obtained for hydroxy-propylmethylcellulose and the agrochemical formulation were approximately double and 4-5 times higher than for wood flour respectively.

For all materials studied non ignition behaviour was epitomised by a localised charring around the hot spot accompanied by no or little exothermic activity, see Figure 7. As the power increased so did the extent of charring around the hotspot surface. In the sub critical test the extent of charring was often significant.

In the case of wood flour and carbon critical behaviour was epitomised by a fully propagating combustion/ decomposition, charring of the entire sample, significant volume loss and measured sample temperatures well in excess of 500°C, see Figure 8.

The hydroxypropylmethylcellulose exhibited a partial propagating combustion/decomposition, which was accompanied by exothermic activity (exotherm  $>60^{\circ}$ C), as was the case with the agrochemical formulation. However, for the agrochemical formulation if a large enough power was applied a full propagating combustion/ decomposition was initiated.

# CRITICAL TEMPERATURE

The experimental control parameter was power rather than surface temperature as this presents additional complexity within the control system. However, the experimental surface temperature was measured and was shown to provide a slight improvement with regard to reproducible

Table 2. Lowest no ignition temperature for each test material and hotspot size studied

	No ignition temperature/(°C)						
Hotspot diameter/(mm)	Wood flour	Carbon (Norit)	Hydroxy	Agrochemical formulation			
6	232	238	272	340			
9	253	205	267	377			
14	218	187	258	310			
16	267	280	255	337			
20	320	180	268	314			

0.125

1

	Sub critical – Critical power/(W)					
Sample volume/(l)	Wood flour	Carbon (Norit)				

3.96 - 4.10

3.53-3.83

3.92 - 4.12

3.35 - 3.66

 Table 3.
 Sub critical – critical power for each test material and sample volume studied

**Table 4.** Average temperature for each hotspot size studied inKieselguhr (3W applied)

Hotspot diameter/(mm) (No. of hotspots studied)	Temperature (°C			
6 (7)	413			
9 (9)	318			
14 (6)	250			
16 (4)	219			

values. Therefore, it has been used as an additional characterising parameter.

As with the power values the critical boundary in wood flour was not clearly defined for each of the hotspot sizes, see Figure 6, but the no ignition/ignition regions were once again clearly established.

The no ignition data presented in Table 2 shows for a given size of hotspot the critical temperature, as defined by the lowest no ignition temperature, is different for all of the materials studied. However, unlike with power a consistent pattern for the 4 materials studied was not observed. The critical temperature in the agrochemical was always the highest though, the value for the 20 mm hotspot compared with wood flour being the only exception.

The no ignition data for an individual material across the hotspot size range is fairly well grouped. In



Figure 7. Sub critical behaviour depicted by a localised charring around the hotspot

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hydroxypropylmethylcellulose it is very tightly grouped ( $264^{\circ}C 69^{\circ}C$ ). In wood flour if the 20 mm hotspot temperature value is excluded (only one 20 mm hotspot was used and it is likely the no ignition value is at the top end of the expected range), the remaining data is tightly grouped ( $242^{\circ}C 625^{\circ}C$ ), as is the data for the agrochemical ( $344^{\circ}C 634^{\circ}C$ ). This suggests a critical temperature for ignition may exist that is independent of hotspot size for these materials.

The no ignition data for the 14 mm hotspot is relatively low for all 4 materials. No obvious explanation is offered for this observation and it is deemed to be within the range of experimental variability.

In Figure 6 the lowest ignition temperature for each hotspot size is plotted against the relevant no ignition data range. It would be expected that this value would not be lower than the lowest no ignition temperature, but this is not the case with the 14 and 16 mm hotspots. However, the difference is very small (6 and 8 K respectively).

# CRITICAL POWER HOTSPOT SIZE RELATIONSHIP

The lowest critical power data expressed graphically in Figure 9 indicates a potential relationship between applied power and the diameter of the hotspot, i.e. critical power increases with increasing hotspot size. If a critical temperature for ignition exists, a likely explanation for the potential relationship between size and power is the requirement for more power to increase the temperature of the hotspot surface for the larger sizes. The data detailed in Table 4 clearly shows that for the same applied power the hotspot surface temperature is lower as hotspot size increases.

# CRITICAL TEMPERATURE HOTSPOT SIZE RELATIONSHIP

The data presented in Figure 6 and Table 2 suggests no relationship between ignition temperature and size of hotspot.



Figure 9. Relationship between the lowest measured critical power and hotspot size for each material studied

#### SAMPLE VOLUME

Rogers 2006, reports powder heap size has an affect on the power and temperature required for ignition. The data reported in Table 3 supports this conclusion. It can clearly be seen that for both carbon and wood flour the critical power is lower with increased sample volume.

## CONCLUSIONS

A test method has been developed that can be used to determine the critical no ignition/ignition boundary for materials susceptible to combustion/decomposition initiated by a localised hotspot. Although the boundary was not sharply defined for either of the characterising parameters (power and temperature), clear ignition/no ignition regions were established. As a consequence, the test can be used to distinguish between different materials using either power or temperature, some materials requiring a relatively small amount of heat energy to initiate combustion/decomposition (low power, low temperature), others requiring more.

The test method can be used to differentiate between two distinct types of thermal event, i.e. a full propagating combustion/decomposition with extensive fuel consumption and substantial exothermic activity, or a partial propagating combustion/decomposition accompanied by less substantial exothermic activity. A number of tests currently exist that can be used to establish some of the characteristics mentioned above, in particular the layer test (T5) and burning test. Whether the hotspot method is better than these existing test methods is a topic for future study.

The critical power determinations for the 4 materials studied show a potential relationship between critical power and hotspot size. It is possible the relationship is linear, and this fits with published theoretical and numerical tests (McIntosh et al. 2002). The temperature data for 3 of the materials studied suggests a possible critical ignition temperature may exist that is independent of hotspot size.

The sample volume has an affect on critical power, for larger volumes it is lower. This is due to increased thermal insulation within the bulk powder when more material is present.

Additional work is required in a number of areas. In particular sample volume appears to affect critical behaviour but the extent of this behaviour is not clear.

Potentially critical conditions are defined by an onset temperature (as is the case in other standard tests). Further test work should seek to control the surface temperature directly rather than measure it as a secondary parameter.

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