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Methane–air mixtures ignited by CW laser-heated targets on optical fiber tips: Comparison of targets, optical fibers, and ignition delays

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Abstract

Fiber optic systems are being deployed in locations where explosive gas atmospheres are normally present or are present under fault conditions. The National Institute for Occupational Safety and Health, Pittsburgh Research Laboratory (NIOSH, PRL) conducted a study of laser safety in potentially flammable environments. Researchers conducted experiments to estimate the mean and standard deviation of laser powers needed to ignite 6% methane–air atmospheres using single mode optical fiber tips covered by two types of iron oxide (Fe_3O_4 and $(FeMn)_2O_3$) mixed with a ceramic adhesive. The iron oxides, heated by a 1064 nm continuous wave laser, ignited the methane–air mixtures at similar powers. The minimum igniting power and maximum non-igniting power (10 tests) were 407 and 350 mW, respectively, using a 62.5 μm fiber. Laser beams guided by 125 and 80 μm diameter cladding single mode fibers produced similar methane–air igniting powers. Ignition was not observed using coal particles at powers that produced ignition with the iron oxides. Threshold ignition delays using the single mode fiber were approximately proportional to the inverse square of the igniting power. Ignition delays were significantly longer than the reported activation time for a commercial fiber optic power limiter. Comparisons are made with the results of other researchers.
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Keywords: Methane ignition; Non-beam hazards; Fiber optic power limiter

1. Introduction

Fiber optic systems are being deployed in locations where explosive gas atmospheres are normally present or are present under fault conditions. For example, fiber deployment through sewer systems and natural gas lines is occurring to overcome the "last mile" hurdle for metro fiber optic systems (Jeyapalan, 2002). Natural gas consists mostly of methane gas (typically greater than 85%), with variable and much smaller amounts of ethane, propane, other hydrocarbons, and hydrogen. Natural gas line faults produce flammable gas–air mixtures. Natural gas migrating from gas line faults into sewer systems has caused explosions. Sewer systems may contain flammable concentrations of methane–air generated from sewage.

The optical radiation guided by fiber optic systems can, under certain conditions, be considered as a potential ignition source for explosive gas atmospheres. For wavelengths in the visible through mid-infrared spectrum, optical ignitions occur most easily when a target contacts the beam and converts the optical energy to thermal energy. For small beam diameters, the best targets for causing ignition absorb most of the optical radiation, do not vaporize as they become hot (i.e. do not dissipate thermal energy prematurely), and match the dimensions of the beam for maximum heat generation. An optical fiber tip that becomes dirty can form a target approximating these conditions.

One method of preventing optical ignitions is to limit the beam power below levels needed to cause ignition. This method may be most advantageous where the laser beam may be exposed to a suitable target and flammable gas–air mixture under normal or fault conditions. For example, a fiber optic cable within a natural gas line that is ruptured

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Table 1
Minimum igniting optical powers and most easily ignited mixtures reported for methane–air

Study	Optical fiber core diameter (μm)	Target material	Methane–air (vol%)	Minimum igniting power (mW)
Welzel et al. (2000)	62.5	Iron–manganese oxide layer	5	304
Zhang et al. (1992)	50	40 μm Appin coal particle	8	300

accidentally. This single event is likely to produce a flammable gas–air mixture and an exposed optical fiber tip that becomes obstructed by foreign material (e.g. dirt).

Optical equipment faults can produce over-power conditions that may pose an ignition risk. Independent over-power fault protection may be provided by optical devices spliced into the fiber optic system (Marrapode & Oron, 2003). These devices are designed for single mode fiber and require a certain amount of time to activate. Devices that activate before potential targets can heat sufficiently would prevent optical ignitions. The time that it takes to heat a target to cause ignition of the gas is referred to as the ignition delay. Detailed knowledge of methane ignition delays would be useful for designing power limiters capable of preventing ignitions.

The National Institute for Occupational Safety and Health, Pittsburgh Research Laboratory (NIOSH, PRL) conducted a study of laser safety in potentially flammable environments.[1] Estimates of the mean and standard deviation of 1064 nm continuous wave (cw) laser powers needed to ignite 6% methane–air atmospheres using single mode optical fibers and several types of laser targets are reported. Ignition delay times are reported as a function of laser powers and compared to the activation time of a commercial optical power limiter. Comparisons are made with the results of other researchers. Results are applicable where methane gas poses an explosion hazard, such as the fiber-in-gas line and fiber-in-sewer installations.

2. Literature review

Minimum igniting powers for methane–air mixtures reported by Welzel, Schenk, Hau, Cammenga, and Bothe (2000) and Zhang, Hills, Zheng, Wall, and Samson (1992) are listed in Table 1. Target diameters above 62.5 μm required higher powers for ignition than listed in the table. These results were obtained using a 1064 nm laser coupled to optical fibers, the laser beam guided to targets applied to the output tip of the optical fiber placed in the flammable atmosphere. Welzel et al.'s (2000) experiments were conducted in a vessel heated to 52 °C. Not listed in the table are the results of Schenk, Bothe, and Cammenga (2001), who reported no ignitions of methane below 370 mW with mostly combustible targets attached to 62.5 μm core fiber tips. Minimum igniting powers for methane were not reported. The combustible targets included carbon black, anthracite coal, coking steam coal, high volatile coal, and a toner. The toner consisted mainly of ferrite powder on a styrene acrylate copolymer base. The toner left a reddish residue on the fiber tip, presumably *non-flammable* ferric oxide (Fe_2O_3).

Several researchers reported lean methane–air mixtures, ranging from about 5% to 6% methane–air, produced the lowest igniting powers with cw lasers (Dubaniewicz, Cashdollar, Green, & Chaiken, 2000; Hawksworth, 2000; Welzel et al., 2000). One exception was Zhang et al. (1992), who reported 8% as the most easily ignited methane–air concentration. Hertzberg (1989) suggests lean methane–air mixtures ignite most easily because of selective diffusion. The lighter methane molecules enter the combustion zone faster than oxygen molecules, creating a fuel rich mixture in the ignition volume. Lean methane–air mixtures should therefore create most easily ignited stoichiometric mixtures (9.48 vol% (Kuchta, 1985)) in the ignition volume close to the ignition source. Very short duration, high peak power pulsed ignition sources also ignite leaner methane–air mixtures most easily, but at concentrations greater than 6% methane–air. For example, 8.3% methane–air is used as the most easily ignitable concentration for mine apparatus approval testing with electrical sparks (MSHA, 1995). 6% methane–air was used for most ignition tests reported here. One series of tests was conducted using 8% methane–air in an attempt to duplicate Zhang et al.'s (1992) results.

Threshold igniting laser powers generally decrease with beam diameter, but appear to level off for fiber-optic guided beam diameters below about 100 μm (Welzel et al., 2000). This leveling off has been factored into safe power recommendations for optical systems used in flammable environments (ANSI/ISA, 2004; Carleton, Bothe, Proust, & Hawksworth, 2000a; McGeehin, 1994). Welzel et al. (2000) and Schenk et al. (2001) suggest the leveling off may be due to heat loss from the fiber core to the fiber cladding, or may be due to a dimensional property of the heat source comparable to, but smaller than, the quenching distance of electrical sparks. The optical ignition power leveling off correlates with the common 125 μm cladding diameter of the single mode and 62.5 μm fibers used for testing, adding credence to the cladding heat-sinking hypothesis. This suggests that fibers with smaller cladding diameters may produce ignitions with lower powers. In the current study, ignition tests were conducted with commercially available

[1]The findings and conclusions in this report are those of the author and do not necessarily represent the views of the National Institute for Occupational Safety and Health.

80 μm cladding single mode fiber in an attempt to observe any influence of cladding diameter on igniting power. Lower observed igniting powers with the 80 μm clad fibers could justify lower safe power recommendations for optical beams guided by these fibers.

Optical equipment may be installed in locations where potential beam targets are not well known or controlled. For these situations, researchers have taken a worst-case approach in selecting targets for experimental research. Using cw lasers with small beam diameters, strongly absorbing and thermally stable targets appear to ignite flammable gases most easily. Moore and Weinberg (1987) were unable to ignite methane–air mixtures using laser-heated cotton wool particles, which turned to ash and disintegrated. They were able to ignite methane–air mixtures using thermally stable refractory insulating wool as the laser target. The insulating wool absorbed the selected laser wavelength well. Welzel, Bothe, and Cammenga (1994) found black iron manganese oxide $(FeMn)_2O_3$ targets produced the lowest igniting powers of a number of targets tested under most experimental conditions using cw lasers. Schenk et al. (2001) found that a toner consisting mostly of ferrite powder ignited certain other flammable atmospheres most easily, but not at powers below that obtained with the $(FeMn)_2O_3$ targets. Dubaniewicz et al. (2000) found Fe_3O_4 targets produced slightly lower igniting powers than coal targets in methane–air mixtures, using a non-flammable grease to attach the targets to large core optical fibers. Larger coal particles required higher laser powers to ignite the methane–air, suggesting the heat of combustion from the coal was not a significant contributor to ignition compared to the heat generated by the laser. Hawksworth (2000) observed combustible targets burn away without igniting selected gases at power levels that produced ignitions with $(FeMn)_2O_3$. The $(FeMn)_2O_3$ produced lower igniting powers for selected atmospheres when it was mixed with a ceramic adhesive and applied to large core diameter multimode optical fiber tips as a paste (Carleton, Bothe, Proust, & Hawksworth, 2000b). One exception was Zhang et al. (1992), who reported that Appin coal particles ignited methane at the lowest powers.

The $(FeMn)_2O_3$ and Fe_3O_4 absorb well over a range of wavelengths (as reported in the materials section), and so can be heated efficiently with a variety of laser wavelengths. These iron oxides are not flammable, and so do not vaporize or disintegrate easily upon heating. Mixing the iron oxides with a ceramic adhesive should enhance the thermal stability of the laser target. The adhesive should also reduce the likelihood of the target dislodging from the fiber tip during tests. Coal and combinations of iron oxide and ceramic adhesive mixtures were used as targets in the current study as a conservative worst-case approach to targets that may obstruct optical fibers.

Zhang et al. (1992) reported a 45 ms delay time for igniting 8% methane–air using 360 mW to heat a 40 μm Appin coal particle attached to a 100 μm core fiber tip. Although Moore and Weinberg (1987) did not use optical fibers, they observed that methane–air produced longer ignition delays than several other hydrocarbons tested, and ignition delays generally decreased hyperbolically as radiant power flux increased. Dubaniewicz et al. (2000) observed methane–air ignition delays increased with decreasing power, using coal and Fe_3O_4 targets on large core optical fiber tips. Ignition delays also increased with larger targets.

3. Equipment

An IPG Photonics PYL-10 M Ytterbium fiber laser system was used for ignition experiments. The fiber laser wavelength was centered at 1064 nm and the output power was adjustable up to 10 W cw. IPG indicated the overshoot was less than twice the set power, settling to the set power within 10 μs. The laser controller provided a laser-on electrical output signal used for ignition delay measurements. The system combines a red aiming laser with the high power beam within a single mode fiber optic pigtail. The pigtail was fusion spliced to a Corning single mode 1 by 2 coupler to protect the laser from fiber fuses. Power levels were also kept below 1 W to avoid possible fiber fuses. A Scientec power meter (model D200PC) with attached calorimeter (model AC2500) measured the laser powers. Scientec checked the meter accuracy annually, traceable to National Institute of Standards and Technology (NIST) standards. Power measurements are reported to be accurate to within $\pm 5\%$.

Single mode fibers used for experiments included a 9/125 (core/clad) μm diameter Corning SMF-28 silica fiber, and a 6.6/80 μm 3 M silica fiber. These single mode core diameters are the mode field diameters at the 1300 or 1310 nm wavelength. More than one mode propagates through these single mode fibers at the 1064 nm wavelength. Other fibers used for experiments included a 62.5/125 μm Corning multimode silica fiber and a 1500/1650 μm Fiberguide Industries AFS silica fiber. The single mode and 62.5 μm fibers were fusion spliced to one of the couplers output ports. SMA connectors were used to couple the laser beam to the 1500 μm fiber. A fiber optic cleaver (York model FBK 11C) provided a flat, perpendicular, optical surface on the single mode and 62.5 μm core fiber tips. A manual cleaving tool was used for the 1500 μm fiber.

Ignition experiments were conducted in a 20 L test chamber (Fig. 1) designed for explosion testing of dusts, gases, and hybrid mixtures (Cashdollar & Hertzberg, 1985). The chamber can be used at initial pressures that are below, at, or above atmospheric as long as the maximum explosion pressure is less than 21 bar, which is the rated pressure of the chamber. Sapphire windows allowed viewing inside the chamber. The concentration of the gas–air mixture was determined by partial pressures. The chamber pressure was set to 1 bar at room temperature. A fan within the chamber provided mixing. The chamber instrumentation included a pressure transducer

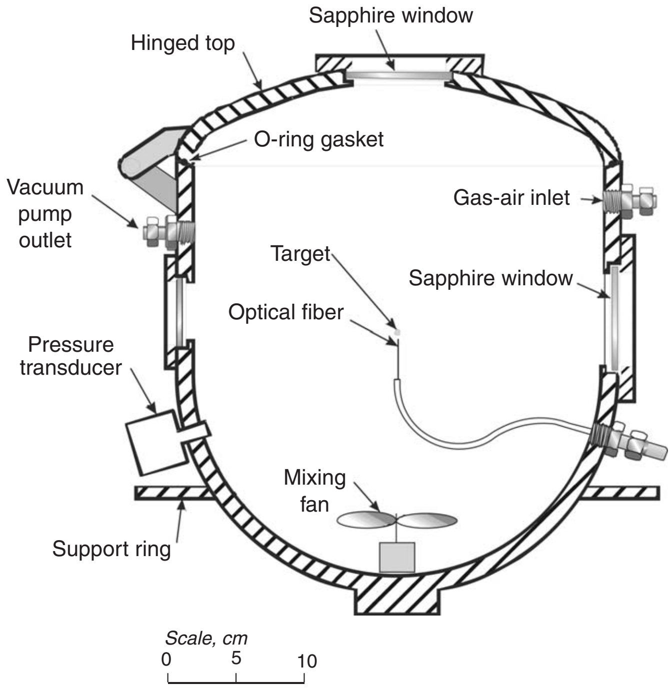

Fig. 1. A 20 L chamber laser ignition test setup.

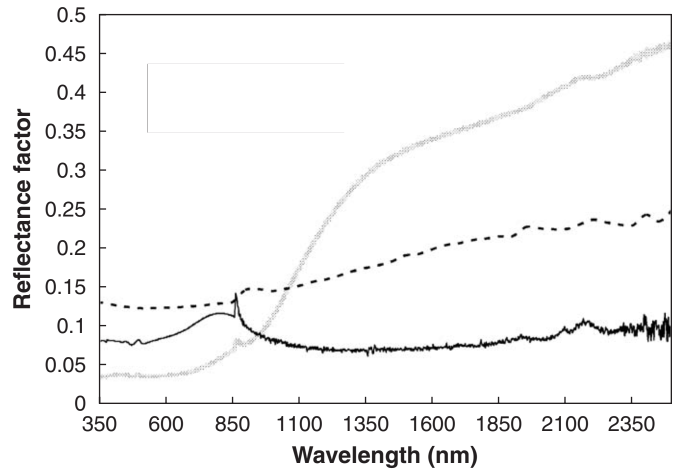

Fig. 2. Reflectance of Pittsburgh coal, black iron oxide and Bayferrox 303 T from 350 to 2500 nm.

(Viatran model 218-28). A personal computer (PC) based data acquisition program recorded the pressure transducer signal and the laser-on signal from the laser controller. A high-speed video camera (NAC model 400) recorded some of the tests.

4. Target materials

Two types of iron oxide were used as laser target materials. Bayferrox 303 T is a mixed iron manganese oxide ($(FeMn)_2O_3$) with a 0.6 μm particle size. Black iron oxide is a combination of ferrous (FeO) and ferric (Fe_2O_3) oxide having a theoretical formula of Fe_3O_4 and average particle size of 0.4 μm. These iron oxides are non-flammable.

The coal dust was Pittsburgh seam, high volatile bituminous coal mined from the NIOSH PRL Safety Research Coal Mine. The volatility is 37% and ash content is 6% (typical) (Cashdollar, 1996). The coal dust was sieved to produce 38–45 μm particles.

Labsphere Inc. measured reflectance factors for the target materials over a spectral range from the ultraviolet to 2500 nm (Fig. 2). In Fig. 2, the black iron oxide becomes a stronger absorber than Bayferrox 303 T for wavelengths above 950 nm. At the 1064 nm wavelength, the absorptance (1−reflectance factor) of the Bayferrox 303 T and coal was 85%, and the absorptance of the black iron oxide was 92%. Other researchers reported higher coal dust absorption at the 1064 nm wavelength (Schenk et al., 2001). The coal dust sample sent to Labsphere consisted of particles significantly larger than the iron oxide samples, and reflecting facets could be seen on many coal particles under the microscope, probably increasing the reflectivity of the coal sample relative to the iron oxides. All three materials absorb well at the 1064 nm wavelength.

For most tests the iron oxides were mixed with a Zirconia based ceramic adhesive (Cotronics Resbond 904), 20% iron oxide to 80% adhesive by weight. The white adhesive mixed with the iron oxides produced a gray colored mixture, likely absorbing the laser less efficiently than the iron oxides alone. The Resbond 904 provides adhesion up to 2200 °C.

5. Methods

An adequate length of fiber was pulled through a feed-through in the 20 L chamber (Fig. 1) to allow preparation of the fiber tip. A fiber optic cleaver (York model FBK 11C) provided a flat, perpendicular, optical surface for each ignition test. The power emanating from the cleaved end of the fiber was measured before and after the test. For tests with the Resbond 904 adhesive, a thin layer of the target paste sufficient to block the red aiming laser was applied to the fiber tip and air dried with the aid of the mixing fan. For tests with no adhesive, the $(FeMn)_2O_3$ was mixed with ethyl alcohol and applied to the fiber tip as a thin layer until the aiming laser was blocked. The $(FeMn)_2O_3$ alcohol mixture was air dried before proceeding, presumably leaving only the $(FeMn)_2O_3$ on the fiber tip. For the coal dust experiments, the particles were placed on the fiber tip by pushing the fiber along a dish containing the particles. Blockage of the aiming laser indicated the coal covered the fiber tip. Single mode fiber was used for the coal particle tests in an attempt to ensure that most of the laser beam was absorbed by the particles.

The fiber was placed in the chamber pointing upward. The chamber was evacuated, and methane–air concentrations were set using partial pressures. The mixture was allowed to settle for about 1 min after fan mixing. For tests with no adhesives, researchers looked through the window at the top of the chamber to ensure the aiming laser was not visible, indicating the target was still attached after mixing. Ignitions produced over-pressures of greater than 3.3 bar and non-ignitions produced over-pressures of less than 0.05 bar. Non-ignitions were generally repeated 10 times before ending a test series (Table 2).

The ignition delays were measured from the PC data plots of the laser-on signals, pressure traces, and rate of pressure rise (dP/dt) traces versus time. High speed video recordings of selected tests showed the flame front emanating from the fiber tip about 56 ms before an appreciable rate of pressure rise in this chamber. Ignition delay times reported here based on the pressure transducer measurements were reduced by 56 ms to account for the difference. Microsoft Excel 2002 was used to calculate data trends and coefficients of determination (R^2).

Researchers used the Bruceton Up and Down method (Dixon & Massey, 1983, Chapter 19; Dixon & Mood, 1948) to estimate the igniting power mean and standard deviation. The mean igniting power is the power level that has a 50% probability of igniting the gas. An initial laser power was chosen for an ignition test. If the test resulted in an ignition, the laser power was decreased by 50 mW for the next test. The laser power was increased by 50 mW for a subsequent test when a test resulted in a non-ignition. After each series of Up and Down tests were completed, the number of ignitions and non-ignitions were counted separately, and the group that produced the lower tally was used for the statistical analysis.

The Up and Down method requires several experimental conditions be met, and these conditions may not be known beforehand. The method requires the variate to be approximately normal near the mean. Prior to testing, researchers chose to use the igniting power itself as the variate. Shapiro-Wilk tests, normal probability plots, and boxplots were used to assess the distributions. The single mode fiber distributions were accepted as being approximately normal near the mean. The Up and Down method also requires the testing step to be less than 2 standard deviations to simplify the statistical analysis. Researchers chose 50 mW as the step power prior to testing, and subsequently this choice was also accepted as satisfying the method requirements.

6. Results

Table 2 summarizes results of tests to determine which targets produced the lowest methane–air igniting powers. Threshold igniting power is the lowest igniting power observed under specified test conditions. The tests using Pittsburgh seam coal and no adhesive (Table 2, row 1) were an attempt to duplicate Zhang's reported 300 mW ignition (Table 1), but with single mode fiber and Pittsburgh coal. No ignition was observed in 10 attempts using 310 mW. The power was doubled to 620 mW and, again, no ignition was observed in 10 attempts. The coal particles burned off or fell off the tip during heating without igniting the gas. The 5 W threshold igniting power for the 1500/1650 μm fiber tip blocked by the iron oxide–ceramic adhesive mixture is the same as that reported by Hawksworth (2000). The iron oxides mixed with the ceramic adhesive ignited the methane–air at lower powers than with no adhesive, in agreement with results reported by Carleton et al. (2000b). The iron oxide–ceramic adhesive mixtures were therefore selected as a conservative worst case target for the more extensive Up and Down tests.

Table 3 lists mean igniting powers and standard deviations obtained using the Up and Down method. The laser heated the targets to incandescence in all tests,

Table 2
Methane–air threshold igniting powers

Optical fiber core/clad (μm)	Target	Methane–air (%vol)	Threshold igniting power (mW)	Igniting power density (W/mm^2)	Maximum non-igniting power (10 tests) (mW)
9/125	Pittsburgh coal	8	—	—	620
9/125	$(FeMn)_2O_3$	6	518	8150	470
6.6/80	$(FeMn)_2O_3$-Resbond 904	6	431	12600	380
9/125	Fe_3O_4-Resbond 904	6	417	6560	380
62.5/125	$(FeMn)_2O_3$-Resbond 904	6	407	133	350
1500/1650	Fe_3O_4-Resbond 904	6	5000	2.83	4500

Table 3
Up and Down test results for igniting 6% methane–air mixtures

Optical fiber core/clad (μm)	Target	n	Mean igniting power (mW)	Standard deviation (mW)
9/125	Fe_3O_4-Resbond 904	23	535	127
9/125	$(FeMn)_2O_3$-Resbond 904	24	498	80
9/125	Combined iron oxide series	47	516	113
6.6/80	$(FeMn)_2O_3$-Resbond 904	40	521	115

ignition or no ignition. For many non-ignitions, the incandescence ceased or decreased significantly within a few seconds. The aiming laser was visible after many of these non-ignitions, indicating the target was no longer attached or partially attached. As reported in the Discussion section, a T-test indicated the Fe_3O_4 and $(FeMn)_2O_3$ probably produce similar mean igniting powers. Based on this assumption, and being able to combine the 9/125 data to form one continuous Up and Down series, the mean and standard deviation of the combined iron oxide data were also listed in Table 3.

The Up and Down tests provided most of the ignition delay data. Fig. 3 shows ignition delays obtained using the single mode fiber and all targets. Ignition delays using the single mode fibers were quite variable, ranging from less than 0.1 s to over 30 s. Fig. 3 shows only data for ignitions that occurred before 1 s. An approximately inverse square curve fit reasonably well ($R^2 > 0.96$) through the shortest ignition delay (t) measured at each power level (p). These threshold data are listed in Table 4. Zhang et al.'s (1992) 45 ms delay for igniting 8% methane–air using 360 mW to heat a 40 μm particle on a 100 μm fiber is also shown in Fig. 3.

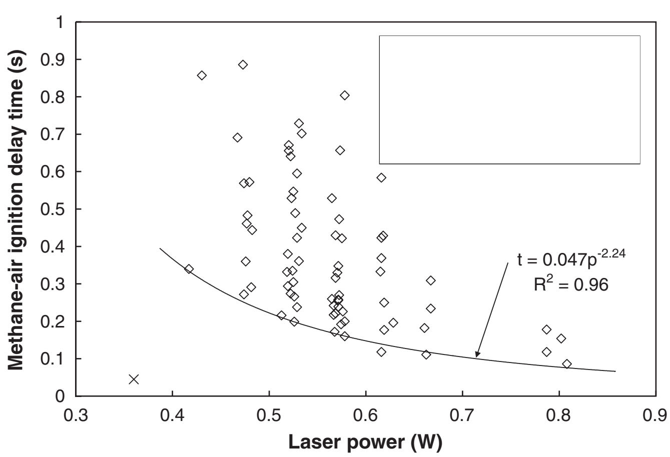

Fig. 3. A 6% methane–air ignition delay vs. laser power using Fe_3O_4 and $(FeMn)_2O_3$ targets attached to single mode fiber tips with or without a ceramic adhesive. Curve fit to threshold delay (t) vs. igniting power (p), and coefficient of determination (R^2) also shown.

7. Discussion

Zhang et al.'s (1992) reported 45 ms delay is much shorter than the delay times reported here (Fig. 3 and Table 4). Some aspects of Zhang et al.'s (1992) result are more indicative of high peak power pulsed ignition possibly caused by excessive laser overshoot. The maximum laser power setting was required to produce methane–air ignitions in Zhang et al.'s (1992) study. This, along with the use of external focusing optics, may have caused significant back reflection. A strong back reflection can cause certain lasers to become wildly unstable (Force Inc., 2005). The surface absorbing calorimeter used by Zhang et al. (1992) may not have had a sufficiently fast response time to detect excessive overshoot.

Zhang et al. (1992) found 8% methane–air to be the optimum concentration using the cw laser. This result is more indicative of high peak power pulsed ignitions. Zhang et al. (1992) also found 8% methane–air to be an optimum concentration using a pulsed laser for additional experiments. The 8% optimum concentration is similar to that used for mine apparatus approval testing with electrical sparks (MSHA, 1995). Other researchers observed lower optimum methane–air concentrations using cw lasers (Dubaniewicz et al., 2000; Hawksworth, 2000; Welzel et al., 2000). Perhaps ignition occurs at 8% most easily with the short duration sparks because diffusion effects described by Hertzberg (1989) don't have enough time to create stoichiometric concentrations with leaner mixtures before the ignition source is removed.

Zhang et al.'s (1992) 45 ms result was obtained using a 22% volatile matter content coal particle target. Schenk et al. (2001) found that high volatile coal targets produced the highest igniting powers and longest ignition delays among a number of targets tested for selected flammable atmospheres other than methane. Schenk et al. (2001) did not observe ignition of methane–air using powers up to 370 mW with any target. Prior research (Dubaniewicz et al., 2000) suggests heat of combustion contributions from the small coal particle should be relatively small compared to the heat generated by the laser, and should not have such a significant impact on ignition delay. Excessive laser overshoot could produce apparently lower igniting powers, shorter ignition delays, and richer

Table 4
Threshold igniting delay times using single mode fiber

Fiber core/clad (μm)	Target	Laser power (mW)	Threshold delay (s)
9/125	Fe_3O_4-Resbond 904	417	0.340
9/125	Fe_3O_4-Resbond 904	475	0.272
6.6/80	$(FeMn)_2O_3$-Resbond 904	527	0.199
6.6/80	$(FeMn)_2O_3$-Resbond 904	580	0.160
9/125	$(FeMn)_2O_3$	618	0.118
6.6/80	$(FeMn)_2O_3$-Resbond 904	664	0.111
9/125	Fe_3O_4-Resbond 904	808	0.086

concentrations of gas that ignite most easily, which is more consistent with pulsed ignition sources.

Another conceivable explanation could be that a smaller target produced the 45 ms ignition. If it could absorb most of the beam, a smaller target would heat more quickly and attain higher temperatures than a larger target. Zhang's particle conceivably could have partially disintegrated prior to ignition, producing a smaller target. Differences in observed ignition delay times are significant enough to warrant independent verification.

The approximate inverse square relationship between threshold ignition delay and power (Fig. 3) is similar to the hyperbolic relationship observed by Moore and Weinberg (1987). One commercial optical power limiter designed to prevent excessive powers in single mode fiber activates within 5 μs under certain test conditions (Marrapode & Oron, 2003). Activation time varies with power. This activation time is significantly less than the ignition delays shown in Fig. 3, listed in Table 4, and reported by Zhang et al. (1992). The power limiting devices may prove to be effective for methane–air ignition prevention. Ignition tests with the devices should be conducted to verify ignition prevention before they are used for such purposes.

A *T*-test for unequal variances was used to compare the mean igniting power of the 9/125 Fe_3O_4-Resbond 904 and 9/125 $(FeMn)_2O_3$-Resbond 904 targets (Table 3). The *T*-test indicated the mean igniting powers could be considered similar at a 0.1 significance level. Any difference in mean igniting power using the two iron oxides was not detected. Using the *T*-test with appropriate assumptions for equal or unequal variances, the 6.6/80 $(FeMn)_2O_3$-Resbond 904 mean igniting power could be considered similar to any of the mean igniting powers for the 9/125 Resbond 904 data (Fe_3O_4, $(FeMn)_2O_3$, or combined iron oxides) listed in Table 3 at a 0.1 significance level. The mean and standard deviation of the combined 9/125 iron oxide-Resbond 904 series appears to converge with the values for the 6.6/80 $(FeMn)_2O_3$-Resbond 904 series. Any heat sinking effects of the cladding material on average igniting powers were not detected. The different types of iron oxide or cladding diameters do not appear to affect igniting powers significantly.

Minimum igniting powers reported by Welzel and Zhang (Table 1) were lower than minimum igniting powers observed in this study (Table 2). The inability to ignite methane at the lower powers or with flammable targets in this study is more in line with Schenk et al.'s (2001) and Moore and Weinbergs's (1987) results. Excessive laser overshoot could produce apparently lower igniting powers than reported here. If it could absorb most of the beam, a smaller target than used in this study would attain a higher temperature and conceivably ignite methane at a lower power. Alternately, the up and down tests suggests 300 mW may be several standard deviations from the mean igniting power, and so more attempts may produce ignition using 300 mW.

Other potential ignition mechanisms should be considered as appropriate. For example, Hawksworth (2001) reported 300 mW readily caused smoldering ignition in combustible coal dust layers, which could possibly lead to large scale burning and subsequent ignition of flammable gas. Power thresholds derived from tests of targets obscuring optical fiber tips suspended in flammable gas atmospheres such as studied here may not be conservative for all conceivable ignition mechanisms.

Heavier hydrocarbons generally ignite at lower powers and more quickly than methane (Moore & Weinberg, 1987). Additional research is warranted to determine ignition delay times of heavier hydrocarbons (e.g. propane) for natural gas that may contain significant amounts of heavier hydrocarbons.

8. Conclusions

For 6% methane–air mixtures ignited by laser heated iron oxide–ceramic targets on single mode fibers tips, the mean igniting power was about 520 mW with a standard deviation of about 110 mW. The igniting power distribution near the mean was approximately normal. The 62.5 μm fiber produced the minimum igniting power (407 mW). No ignition was observed with the 62.5 μm fiber in 10 tests using 350 mW.

Fe_3O_4 and $(FeMn)_2O_3$ targets mixed with a ceramic adhesive and heated by a 1064 nm laser ignited methane–air at similar powers. Ignition was not observed using coal particles without adhesive at powers that produced ignition with the iron oxides.

Using single mode fibers, threshold ignition delays were approximately proportional to the inverse square of the igniting power. Ignition delays were significantly longer than the reported activation time for a commercial fiber optic power limiting device, suggesting such a device may be useful for over power fault protection to prevent methane–air ignition.

Laser beams guided by 125 and 80 μm diameter cladding single mode fibers produced similar mean ignition powers. Heat sinking effects of the cladding material were not observed. The leveling off of threshold powers below the 62.5 μm beam diameter may be comparable to, but smaller than, the quenching distance of electrical sparks. More conservative safe power recommendations for beams guided by 80 μm diameter cladding single mode fibers are not warranted based on these results.

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