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$TiCl_4$ AS A SOURCE OF TiO_2 PARTICLES FOR LASER ANEMOMETRY MEASUREMENTS IN HOT GAS

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SUMMARY

A method of reacting $TiCl_4$ with water saturated gaseous nitrogen (GN_2) at the entrance into a high-temperature gas flow is described. The TiO_2 particles formed are then entrained in the gas flow and used as seed particles for making laser anemometry (LA) measurements of the flow velocity distribution in the hot gas. Scanning electron microscope photographs of the TiO_2 particles are shown. Data rate of the LA processor was measured to determine the amount of TiO_2 formed. The $TiCl_4$ and mixing gas flow diagram is shown. This work was performed in an open jet burner at NASA Lewis Research Center.

INTRODUCTION

The reaction of $TiCl_4$ with water in GN_2 to form TiO_2 was investigated as a method to generate seed particles for high-temperature gas flow in turbine hot section LA measurement applications. This reaction is a simple low cost method to generate large numbers of small TiO_2 particles. $TiCl_4$ reacts with water in air as shown.

$$TiCl_4 + 2H_2O \rightarrow TiO_2 + 4HCl$$

As shown in the reaction equation there is a 4 to 1 ratio of HCl to TiO_2 formed. The total amount is small, however, because small amounts of $TiCl_4$ are used. The HCl could be neutralized, such as with ammonia or sodium bicarbonate as shown.

$$NH_3 + HCl \rightarrow NH_4Cl \text{ (ammonia chloride salt)}$$

or

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$$

Other reactions are also possible. In this experiment we did not attempt to neutralize the HCl.

The general method of particle generation we used was similar to that used by R.R. Craig et al. (ref. 1) to form TiO_2 for ambient temperature LA applications. We used GN_2 as a carrier gas and mixed the $TiCl_4$ gas and water saturated GN_2 at the entrance into the hot gas stream.

The experiment was part of a general investigation of various seed particles that would be suitable for high-temperature applications. Dry TiO_2 and Al_2O_3 (1 μm in diam) have been selected as satisfactory but not ideal for our application. The dry particles were injected into the combustion chamber from a fluidized bed particle generator. Because of the difficulty of breaking up the agglomerates, generally the dry particles were 1 μm or larger. However, large numbers of 1 μm or smaller particles are desired to increase the measurement accuracy of highly accelerated/decelerated flows and turbulent flows. $TiCl_4$ as a source of small particles appeared to be a good selection except for the large amount of HCl formed. In this application, the gaseous HCl formed was not a problem because the exhaust gas was cooled and cleaned by a water spray system directly downstream from the burner.

An open jet burner as shown in figure 1 was used for the experiment (ref. 2). This burner has a wide range of temperature and velocity performance. The fuel/oxidizer used is Jet A and air from NASA Lewis Research Center's central air supply system. The temperatures in the combustion chamber run up to 1426 °C which is below the melting point of Al_2O_3 and TiO_2. LA measurements of the exhaust flow velocity distribution were made by an LA fringe system that is used for development of LA hardware and software systems for high-temperature applications. The total system is automated (ref. 3).

The injection system used is shown schematically in figure 2. We decided to use a dry GN_2 carrier gas for the $TiCl_4$ to prevent particle formation in the feed line. The feed line was 3/8-in. i.d. The $TiCl_4$ was heated to increase the vapor pressure, but the temperature was not controlled. Additional GN_2 also was run through a water bath to pick up moisture and then mixed with the $TiCl_4$ in a concentric mixer inside the burner. Using the GN_2 instead of air to carry the water vapor was not necessary; it was just more convenient to use one source of gas. In the concentric mixer, the $TiCl_4$ gas was the inside tube and the water vapor was in the outside which extended 1 in. beyond the inside tube. This allowed mixing to form particles inside the burner but protected them from the flame until mixed. The water bath insured sufficient water in the GN_2 to react to all of the $TiCl_4$ being injected.

TEST RESULTS

During test runs to evaluate particle generation, the $TiCl_4$ carrier gas pressure was set to 48 N/m^2 (7 psig) and the GN_2 moisture carrier gas pressure was set to 10 N/m^2 (1.5 psig). This mixture gave a 50 kHz data rate as measured on an LA data processor. The temperature of the burner gases was measured at 850 °C. Primary and secondary air pressure to the burner were 255 N/m^2 (37 psia) and 138 N/m^2 (and 20 psia), respectively. During a period of approximately 30 min, the data rate was varied from 1 to 50 kHz by adjusting the $TiCl_4$ carrier gas pressure and the water carrier gas pressure. Since the $TiCl_4$ container was loaded with only about 100 ml of liquid, the amount of $TiCl_4$ gas picked up varied significantly with pressure. Since this was a test configuration to determine feasibility of the method, no attempt was made to improve the performance; however, it was evident that several improvements (listed below) could be made if long term use of this method were desired.

PARTICLE SIZE

TiO_2 particle samples were collected on polished stainless steel plates inserted into the hot exhaust stream approximately 30 cm downstream from the burner outlet. Exhaust temperature at the outlet was 850 °C and the velocity was approximately 300 m/sec. The plates were held in the exhaust at least 30 sec. For analysis the sample plates were coated with 100 Å of gold to increase particle contrast. Scanning electron microscope photographs of the plates are shown in figures 3(a) and (b). Figure 3(a) shows a fairly uniform covering of small particles and also some TiO_2-coated large carbon particles. The SEM photograph of an area of small particles is shown in figure 3(b). This photograph shows large numbers of particles 1 μm and smaller.

CONCLUSIONS

Large numbers of small particles can be formed by mixing $TiCl_4$ and water-saturated GN_2 at the entrance to a hot gas flow. The HCl also formed would need to be neutralized for most applications. In applications where the HCl can be tolerated or neutralized, the use of $TiCl_4$ is a good source of high-temperature particles for hot flow LA measurements.

Some advantages of using $TiCl_4$ are: (1) large number density of particles, (2) small size, (3) in-situ generation, and (4) constant rate of generation. Some disadvantages are: (1) toxic source liquid and gas, (2) HCl to neutralize, and (3) necessary use of special container materials (glass, ceramic, or stainless steel) for $TiCl_4$. Many improvements could be made to the injection system used in this experiment. Some suggestions are given below.

1. An injector with an HCl neutralizer input downstream from the $TiCl_4$ and GN_2 mixing volume.

2. A heater and temperature controller for the liquid-$TiCl_4$ supply.

3. A controller to maintain a constant pressure differential between the injector gas and the burner pressure.

4. Water purge to clean lines and GN_2 to dry the $TiCl_4$ lines before operation.

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3. Seasholtz, R.G.; Oberle, L.G.; Weikle, D.H.: Laser Anemometry for Hot Section Applications. Turbine Engine Hot Section Technology 1983, NASA CP-2289, 1983, pp. 57-68.

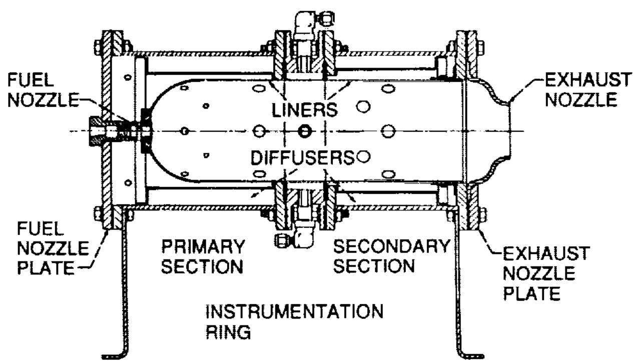

Figure 1. - Cross section of Becon open jet burner.

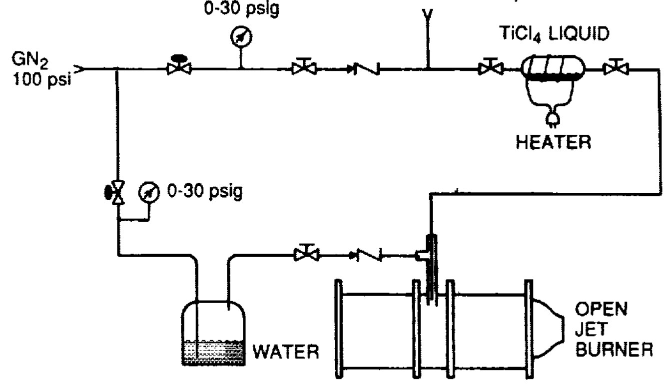

Figure 2. - TiO_2 particle generation system for combusting flows.

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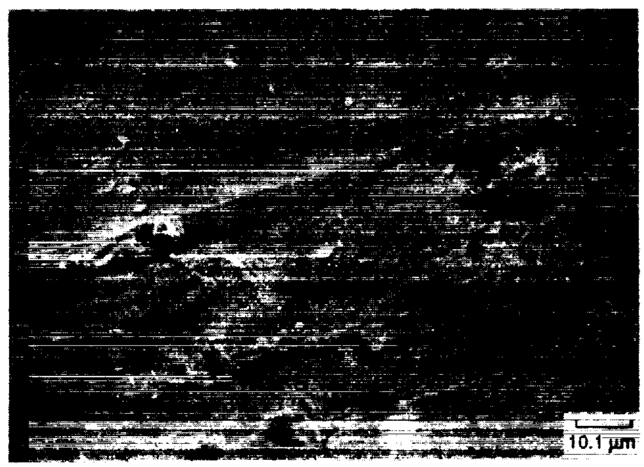
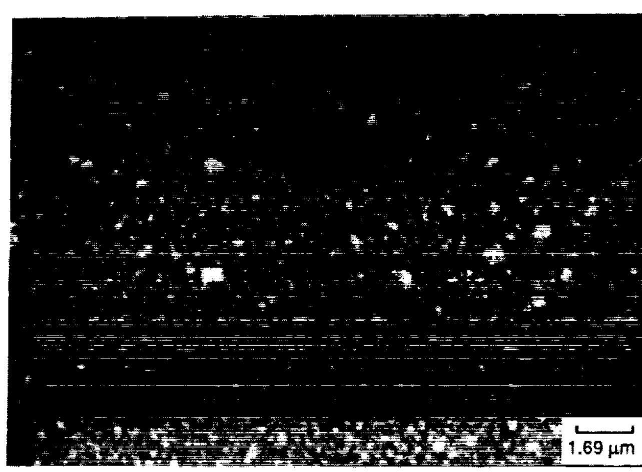


(a) Larger area.



(b) Small area of figure 3(a) at higher magnification to show TiO_2 particles 1 μm and smaller.

Figure 3. - SEM photographs of an area of TiO_2 particles and TiO_2 covered carbon particles on a stainless steel plate.

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