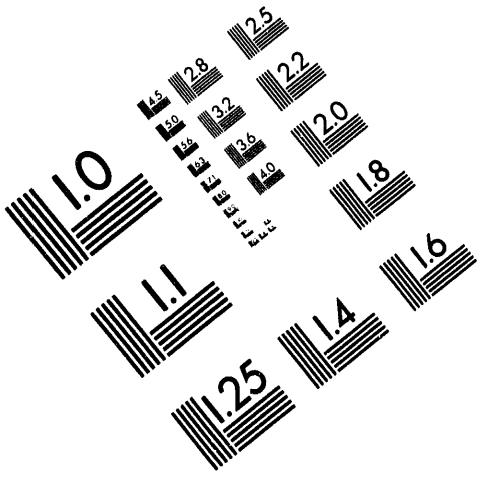

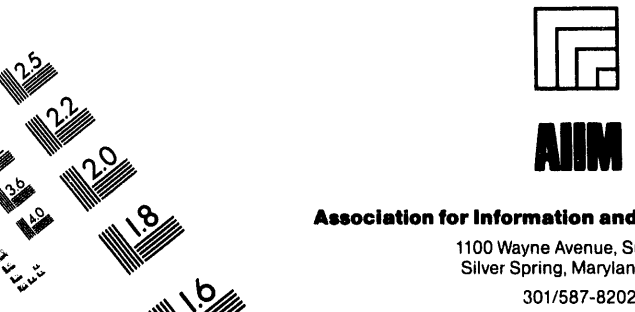

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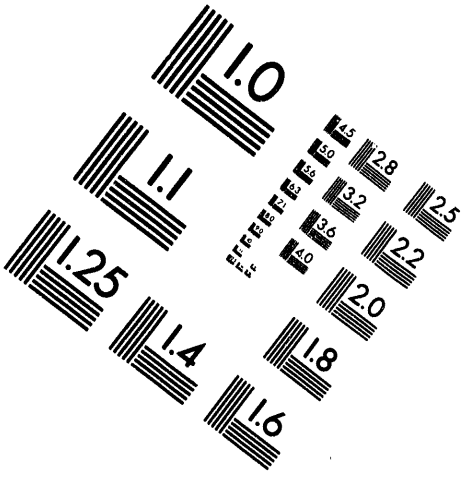

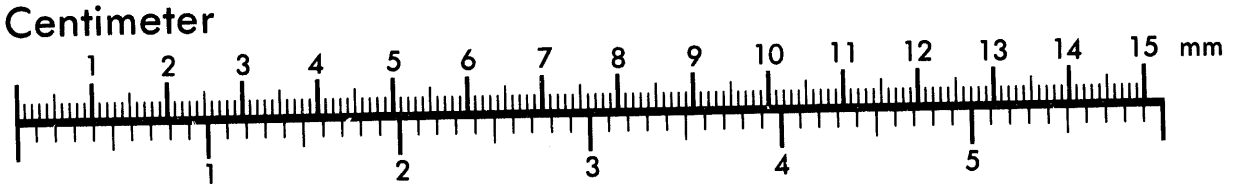

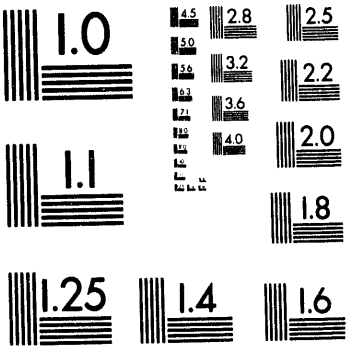

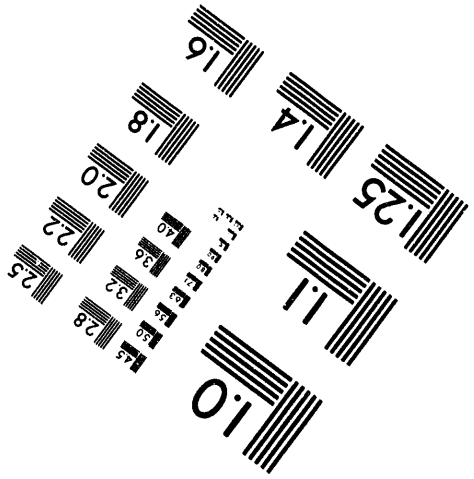

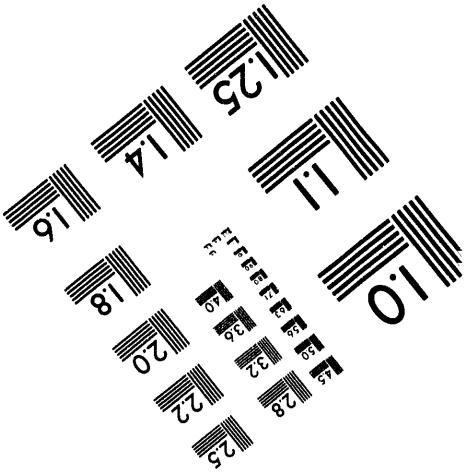

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Report No. FE-MIT-92111-5

Conversion of Light Hydrocarbon Gases to Metal Carbides for Production of Liquid Fuels and Chemicals

Quarterly Technical Status Report for the Period October 1 - December 31, 1993
DOE/PETC-MIT Contract No. DE-AC22-92PC92111

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DISCLAIMER

Submitted to the United States Department of Energy
Pittsburgh Energy Technology Center
Attention: Dr. Arun C. Bose

February 1994

U.S. DOE Patent Clearance is not required prior to the publication of this document.

Abstract

The plasma gun was redesigned, after an accidental burn-out in the previous period, to eliminate any possible arcing pathways outside of the inter-electrode region. A stable Argon arc discharge was obtained during the first test-firing of the completely rebuilt gun. The system flow capacity was determined to be about 3 cfm at ambient conditions. Procurement and installation of process and safety monitoring devices and of components of the sample collection system are in progress. The gas-quenched and water-cooled sample collection probe is fully assembled and is ready for installation. Shakedown-testing of the thermal plasma reactor has also commenced. Debottlenecking work on the gas and cooling water lines was performed to relieve excessive pressure drop through both gas and cooling water systems. For the next period, the shakedown testing will proceed first with the determination of the maximum power levels at which the plasma reactor can be operated at each gas flowrate, with Argon as the plasma-forming gas. The next step will be to complete the assembly of the sample collection system and to check out the entire system to determine other process constraints, first without firing, and then, with firing, still utilizing Argon as plasma gas. Preliminary temperature profile measurements in the cooling chamber will be taken using thermocouples to map out a safe penetration length of the sample collection probe into the chamber. Once the shakedown-testing is completed, the Miller powder feeder will be procured on a trial basis and preliminary scoping runs with methane and CaO and with methane and MgO will be performed.

1. Progress on Task 1: Industrial Chemistry and Applied Kinetics of Light Hydrocarbon Gas Conversion to Metal Carbides, Hydrogen and Carbon Monoxide

1.1 Status of Work as of the End of Previous Reporting Period (July 1 - September 30, 1993)

As of the end of the previous reporting period, machining work on parts of the cooling chamber assembly had been completed and the cooling chamber assembly had been mounted in the fragmentation containment room. A duct leading from the top of this room to the exhasut chimney had also been installed. The cooling water system had been commissioned and the plasma gun system had been mounted in the cooling chamber assembly inside the fragmentation containment room. A test-firing of the arc discharge with Argon conducted in late September unfortunately resulted in a burn-out of some parts of the plasma gun apparently due to arcing outside of the inter-electrode region. However, the damage was considered minor since the major components of the gun, particularly, the anode nozzle and cathode assemblies, remained intact. Only the plasma gun body, made of nylon and phenolic laminate insulation, was damaged severely. Arcing apparently occurred outside the inter-electrode region between the cathode assembly and the gas line, presumably because of insufficient insulation. This incident set back the projected date for completing apparatus construction. It was also decided to redesign the plasma gun body to minimize the possibility of arcing between the cathode and the gas line, by introducing the gas at a point farther away from the cathode assembly.

Further investigation of options for a powder feeder had proceeded, including scale-up of a home-made fluidized bed syringe feeder, used at MIT for delivering coal to a drop-tube furnace, and employment of a mechanical wheel-type feeder sold by Miller Thermal, Inc., of Appleton, WI. The latter feeder had given a promising demonstration run at MIT. The idea of using an Argon plasma as a heat source in carrying out mechanistic studies was also considered. Thus, the plasma reactor, once completed, might be useful for this purpose as well.

1.2 Current Reporting Period: Experimental Equipment Construction

After the burn-out of the plasma gun in late September, the gun was redesigned in October and completely rebuilt by early November. Arcing was suspected to have occurred between the upper part of the cathode assembly A and the metallic gas line B, as illustrated in Figure 1. To eliminate this possible arcing pathway, the gun was redesigned so that the gas line enters the side of the nylon body C (Figure 1) perpendicular to the axis of the cathode A, rather than parallel to it as in the original design. New pieces were fabricated to replace the phenolic laminate insulator D and nylon cap E, and extra pieces were machined to serve as spare parts. In addition, since the original nylon body C was damaged only at the top part where the metallic gas line joined it, the original gas entry hole was plugged and a new hole was drilled at the side, perpendicular to the cathode axis. With these modifications, the original nylon body now serves as a spare part for component C.

A successful test-firing of the gun with Argon as the plasma gas was conducted in November. A stable arc was obtained while varying power input from 2.5 to 7.5 kW. The voltage across the interelectrode gap was measured at about 25 V and the current applied was varied from 0.1 to 0.3 kA. The exit gas temperature measured at 4-5 inches from the bottom of the cooling chamber was around 300°C.

A more systematic shakedown testing of the plasma reactor system was then planned using Argon in order to determine process bottlenecks (i.e. maximum power input obtainable at each flowrate, resulting gas exit temperature, maximum gas flowrate). In preparation for this shakedown testing, the components of the control console were first upgraded to corrosion-resistant materials (i.e. from copper tubes and brass fittings, gauges, and regulators to stainless steel materials). After initial calibration runs of the gas rotameter on the control console, the system flow capacity was determined to be about 3 cfm at ambient conditions, which is around 150% of the capacity of Kim's (1977) *et al.*(1979) thermal plasma system. However, this flowrate required a rather high supply pressure of 80 psi at the rotameter on the control console compared to the 30 - 40 psi supply pressure at which the plasma gun was previously operated. The significant pressure drop through the gas tubing prompted upgrading from 1/4" to 3/8" gas lines. This debottlenecking step also required some modification on the nylon piece C (Figure 1) of the plasma gun to accommodate the larger gas inlet line. After the modifications, a flowrate slightly higher than 3 cfm can now be delivered at a pressure of 60 psi. The only remaining constriction in the system is the 1/8" I.D. by 1/4" long orifice inlet F (Figure 1) to the inter-electrode region. Should there be a need for a higher flowrate, this orifice can be enlarged as well.

When the plasma gun was disassembled to make a larger gas inlet provision on the nylon piece C, the Macor® ceramic disks G and H (Figure 1) on the aluminum adapter and steel flange were found to have melted slightly after the test-firing. These pieces confine the plasma jet within a cylindrical channel I and thermally insulate the aluminum adapter and the steel mating flange (Figure 1) from the end of the plasma jet. The central orifice on the disk G was increased from 3/8" to 3/4" and that on disk H from 3/8" to 1" in order to minimize jet impingement on the walls of the ceramic.

The gas-quenched and water-cooled sample collection probe, designed by Mr. Modestino, has been assembled and leak-tested in stages. In the course of brazing some probe pieces together, several water and gas leaks developed but were subsequently corrected in the probe hardware. Assembly of the sample collection probe was completed during the period and it is ready for installation once the other parts of the sample collection system are in place.

Installation of various process and safety monitoring devices on the reactor system was undertaken. A digital temperature indicator was provided for the thermocouples installed on the outlets of all cooling water lines. In September, flow rate measurements on these cooling water lines were taken in order to size flow switches. These flow switches have been mounted on the cooling water outlets as part of a safety interlock system. A check-out of the cooling water system prior to the test-firing of the gun revealed that excessive back-pressure existed in the drain

header, limiting the flow through some of the smaller lines. A parallel drain header was temporarily installed to reduce the back-pressure and ensure adequate flow through all cooling water drain lines during the test-firing. A dedicated cooling water drain line for the sample collection probe was then installed to relieve the excessive back-pressure in the original drain header. However, a checkout of the newly configured cooling water system showed a further limitation on the supply side. The cooling water requirements of the plasma gun and of the probe cannot be supplied by the same source. Cooling water for the probe will have to be supplied by another source.

The MIT Safety Office requires an air flow of about 1000 cfm at the bottom of the fragmentation containment room which houses the reactor system to ensure adequate ventilation and dilution of the exhaust gases. Currently, the flow is 1000 cfm at the top of the room but is only around 800 cfm at the bottom due to entry of air through gaps and openings along the length of the room. In preparation for firing the system with methane and CaO/MgO in the near future, further sealing of the fragmentation containment room was undertaken.

Finally, procurement and installation of various components of the sample collection system are in progress.

2. Progress on Task 2: Mechanistic Foundations for Converting Light Hydrocarbon Gases to Metal Carbides, Hydrogen and Carbon Monoxide

There was minimal activity under Task 2 in this quarter, as the construction of the thermal plasma reactor is nearing completion and took precedence. The current plan is to utilize the thermal plasma reactor to generate elevated temperature (2000 - 3000°C) gas flows for studies of thermal (i.e. non-plasma) conversion of CH_4-CaO and CH_4- MgO mixtures to CaC_2 and Mg_2C_3, respectively. In this approach, the effluent gas from an argon plasma would be utilized to rapidly heat up initially cool CH_4-CaO and CH_4- MgO mixtures. Proper attention to contacting chamber design and post-contactor quenching is expected to enable this technique to furnish kinetic data.

3. Future Plans

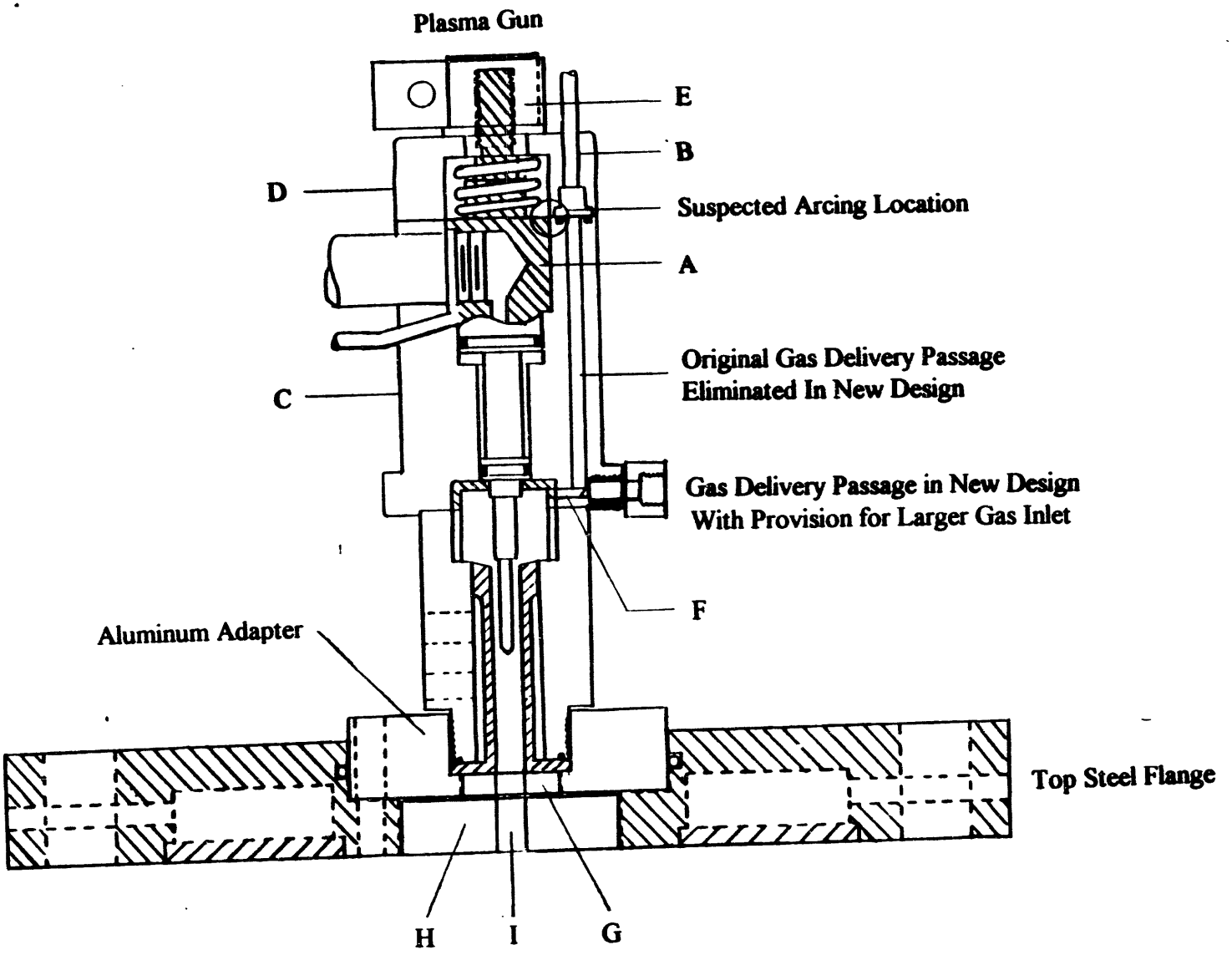
The plan for upcoming work is to proceed with shakedown testing of the system. The maximum power levels at which the plasma reactor can be operated at each gas flowrate will first be determined with Argon as the plasma-forming gas. The next step will be to complete assembly of the sample collection system and to check out the entire system to determine other process constraints (i.e. gas flow through the sample collection system, pressure in the cooling chamber, cooling capacity, exit gas temperature), first without firing, and then, with firing, still utilizing Argon as plasma gas. Preliminary temperature profile measurements in the cooling chamber will be taken using thermocouples to map out a safe penetration length of the sample collection probe into the chamber. Debottlenecking work will be done, as needed. Once the shakedown-testing is completed with Argon, the Miller powder feeder will be procured on a trial basis and preliminary scoping runs with methane and CaO/MgO will be performed.

4. References

1. Kim, C.S., "Formation of CaC_2 from CaO and 'Nascent' Carbon Species in a Rotating-Arc Reactor", Sc.D. Thesis, Department of Chemical Engineering, MIT, Cambridge, MA, (1977).

2. Kim, C.S., R.F. Baddour, J.B. Howard and H.P. Meissner, "CaC_2 Production from CaO and Coal or Hydrocarbons in a Rotating-Arc Reactor", *Ind. Eng. Chem. Process Des. Dev.* **18**, 323-328, (1979).

Figure 1. Schematic Representation of Plasma Gun Internals and Mounting Connections



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