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INDUCTION PLASMA CALCINING OF PIGMENT PARTICLES
FOR THERMAL CONTROL COATINGS

by

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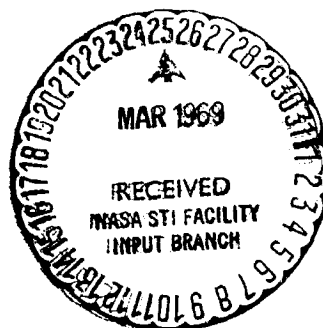
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I   INTRODUCTION AND PROGRAM OBJECTIVE

Several inorganic materials with high reflectance over a wide range
of the solar spectrum are potential pigments for thermal-control coatings
on space vehicles. The most promising current materials are zinc oxide
(ZnO) and zinc orthotitanate (Zn_2TiO_4), but both of these pigments lose
reflectance in the near infrared region during space flight or space
flight simulations because of ultraviolet radiation in vacuum.[1]

Previous research has led to changes in the processing of these pig-
ments that increase their resistance to damage from ultraviolet radiation.[2]
Alkali silicates were found to be beneficial in zinc oxide paints. Later,
an alkali silicate wash coating on the zinc oxide pigment followed by a
low temperature calcination of the treated pigment before paint formula-
tion was found to be more beneficial. Calcination at 650°C fo 1.6 hours
is typical. Rapid bulk diffusion of the alkali silicate in the zinc oxide
particle occurs at 1100°C and must be avoided.

Zinc orthotitanate is formed by a solid-state diffusion reaction be-
tween intimately mixed zinc oxide and titanium dioxide powders.[3] This
reaction is typically carried out at temperatures between 900 and 1100°C.
It has been observed that zinc orthotitanate is more resistant to ultra-
violet radiation damage when higher reaction temperatures are used. How-
ever, the higher temperatures cause sintering of the pigment into unac-
ceptably large agglomerates, which must be subsequently comminuted. The
grinding step evidently introduces point defects into the zinc orthotita-
nate crystal, making it more susceptible to ultraviolet damage.

The normal method for calcining zinc orthotitanate and silicated
zinc oxide involves loading the material in a combustion boat, where the
particles are contiguous and agglomeration by sintering is promoted. If
fine silicated zinc oxide particles or ground zinc orthotitanate particles

could be dispersed within a gas phase during heat treatment, then sintering could be avoided and fine pigment particles resistant to ultraviolet damage could be obtained. The most rapid method of affecting particle heating would be to disperse the particle in a very hot gas such as a flame. However, this is likely to introduce impurities present in the fuel. A plasma, particularly one generated by high frequency oscillations where electrode contamination is avoided, is a more attractive source of clean hot gas.

The objective of the present study was to determine the potential of employing induction plasma heating techniques for calcining pigment particles used in thermal control coatings. Fine particles of silicated zinc oxide and zinc orthotitanate were dispersed in a carrier gas consisting of mixtures of argon and oxygen and were heated by an induction plasma.

II   PIGMENT MATERIALS

Six lots of zinc orthotitanate and two lots of silicated zinc oxide were supplied by NASA from IIT Research Institute. IIT's processing conditions of these 1-pound lots are shown in Table I. The limited funds available permitted plasma calcining tests to be run on only two of these lots: the silicated zinc oxide (batch A-551) that had been dry ground for 6 hours, and the zinc orthotitanate that had been reacted at 1050°C for 18 hours (samples 1 and 2 in Table I). Excess ZnO in the zinc orthotitanate had been extracted with acetic acid, and the resulting material had been sifted through an 80-mesh screen.

TABLE I

IIT'S PROCESSING CONDITIONS FOR PIGMENTS

SRI Sample Number	Pigment	Firing	Subsequent Treatment
2	Zinc orthotitanate	18 hours at 1050°C	-80 mesh*
3	Zinc orthotitanate	18 hours at 1050°C	Ballmilled 24 hours
4	Zinc orthotitanate	18 hours at 925°C	Ballmilled 24 hours
5	Zinc orthotitanate	18 hours at 925°C	-150 mesh
6	Zinc orthotitanate	18 hours at 925°C, 5 hours at 1050°C	Ballmilled 24 hours
7	Zinc orthotitanate	18 hours at 925°C, 5 hours at 1050°C	-80 mesh
1	Silicated zinc oxide		Ballmilled 6 hours
8	Silicated zinc oxide		-20 mesh (no grinding)

*After firing, all zinc orthotitanate powders were leached with $HC_2H_3O_2$ to extract excess ZnO.

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X-ray diffraction analysis of the silicated zinc oxide showed that the major constituent was hexagonal zinc oxide; however, five weak peaks were observed that could not be indexed to ZnO or any other compound. Optical micrographs of silicated zinc oxide showed the discrete particle size to be from 1 to 2 μ. Fisher sieve analysis of this material indicated an average particle size of 0.4 μ. Electron micrographs indicated particle sizes ranging from about 0.1 μ to large aggregates several microns in diameter.

X-ray diffraction did not show other phases to be present in the zinc orthotitanate samples. These particles appear similar to the zinc oxide particles when examined with a microscope. Fisher sieve analysis yielded the following average particle sizes for the zinc orthotitanate samples:

1050°C, ballmilled	1.14 μ
925°C, ballmilled	0.75 μ
925°C/1050°C, ballmilied	1.22 μ

III   EXPERIMENTAL APPARATUS

The apparatus designed and constructed for plasma calcining pigments
had the following functional components:

1. A powder feeder for injecting pigment into an argon/oxygen
   carrier gas stream.
2. A disperser to break up physical agglomerates of pigments
   before injection into the plasma reactor.
3. A 57-mm-diameter quartz plasma reactor tube.
4. A gas cooling train, which also collected much of the powder
   product.
5. A particle filter.

Provision was made for passing particles through the plasma or for secon-
dary injection of particles downstream from the plasma, and experiments
were done both ways. Experiments were run (1) at greater than 1 atm with-
out any reduced pressure on the filter under-flow; (2) at pressure slightly
greater than 1 atm with reduced pressure on the filter under-flow; and (3)
with reduced pressure on the filter under-flow and a vent to discharge ex-
cess gas and thereby maintain the reaction pressure at 1 atm. Results were
reasonably good with any of these procedures, but operation at 1 atm with
an excess gas vent was the most convenient method. Fig. 1 shows a sche-
matic drawing of the apparatus with downstream pigment injection and a
discharge vent to maintain the reaction pressure at 1 atm. Four flow
meters were used to independently proportion the oxygen/argon gas mixture
and regulate the flow rate through the plasma sheath and through the down-
stream injection port. Because of limitations on plasma stability, a
greater fraction of oxygen could be used if most of the oxygen was in-
jected through the downstream port. Pressures and temperatures were moni-
tored on the effluent side of the reaction chamber.
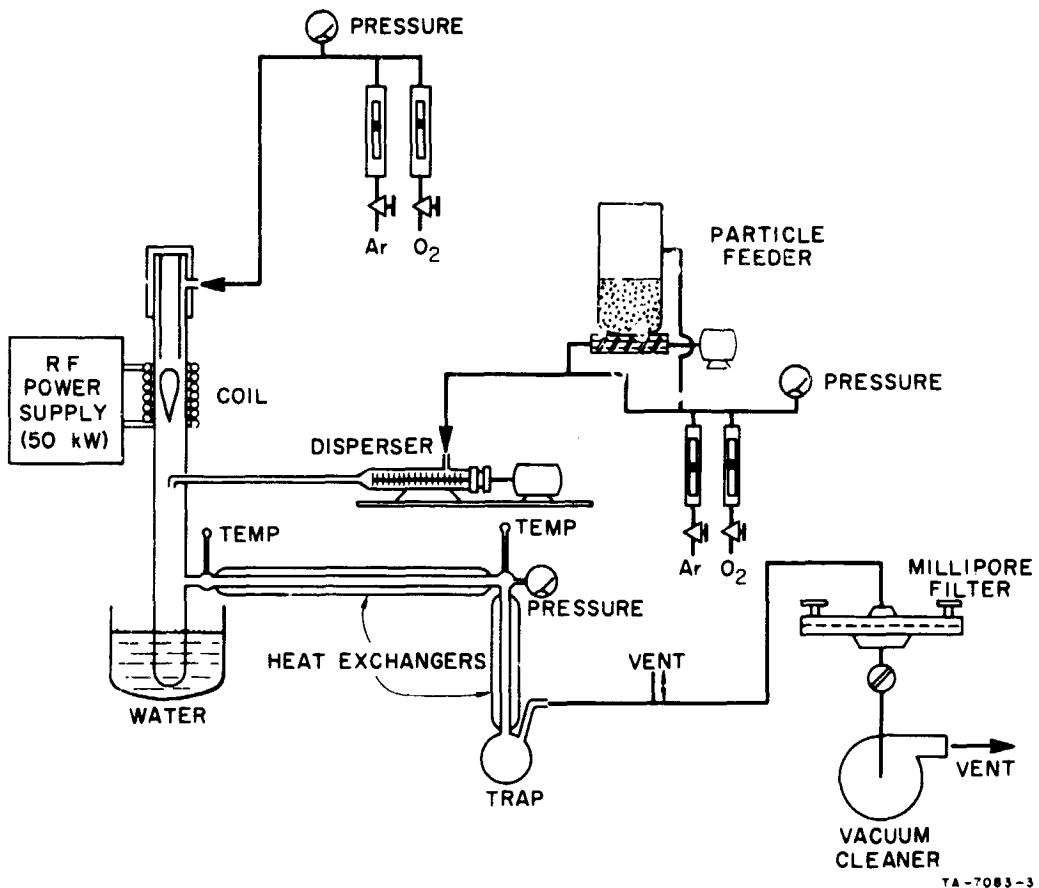
FIG. 1   DIAGRAM OF PLASMA CALCINING APPARATUS
WITH PIGMENT INJECTION DOWNSTREAM

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1. Powder Feeder

An auger-vibratory type variable-speed particle feeder, manufactured by Sylco Corporation, with pressure equilibration of the particle reservoir was employed. Particle feed rates were calibrated against the power setting of the variable-speed drive motor using silicated zinc oxide. The calibration curve is shown in Fig. 2. A feed rate of 1 g/min was used in subsequent plasma calcining experiments. Although the zinc orthotitanate was not calibrated, it was assumed from the comparative density and particle size that the zinc orthotitanate feed rate was similar to the silicated zinc oxide feed rate and the same power settings were used for both. The particle feeder is shown with other parts of the apparatus in Fig. 3.

2. Particle Dispersion

Break-up and dispersion of particle aggregates in the gas stream before they enter the plasma reactor was recognized as one of the major problems at the beginning of this investigation. Two methods of dispersing the particles were investigated: (1) dispersion by a high velocity gas jet coupled with a circulating load of the gas/solid mixture to pick up additional powder from the particle feeder, and (2) dispersion within a cylindrical chamber by a rotating brush. The latter technique was developed by Bradley and Stoddard[4] at Los Alamos. Judging from the light scattered by dispersions, the rotating brush device was the most effective disperser and was used in subsequent experiments.

The brush disperser housing was constructed of brass with a convergent nozzle at one end for the outlet and a side entry port. The apparatus was lined with Teflon after the initial neoprene lining was found to contaminate the pigment particles. A common bottle brush was used; its axis was concentric with the cylindrical housing and was attached to a rotary shaft which passed through a Wilson seal and was driven by the variable speed air turbine motor. Two stainless steel trip bars were attached to the inside wall of the housing.
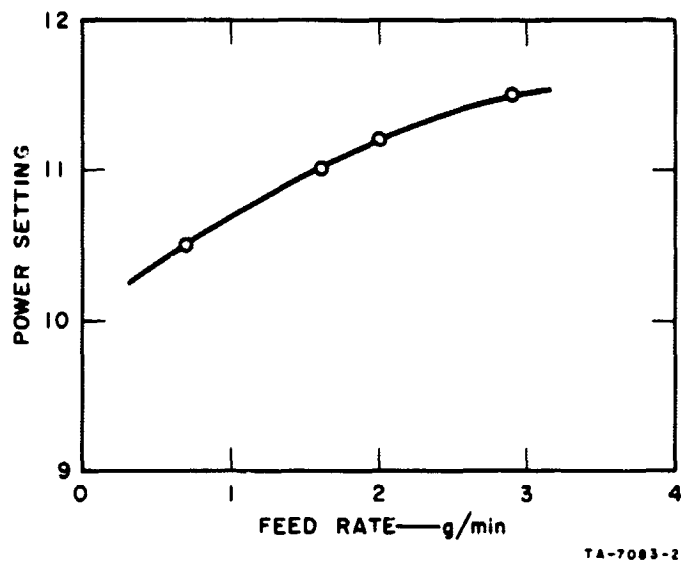
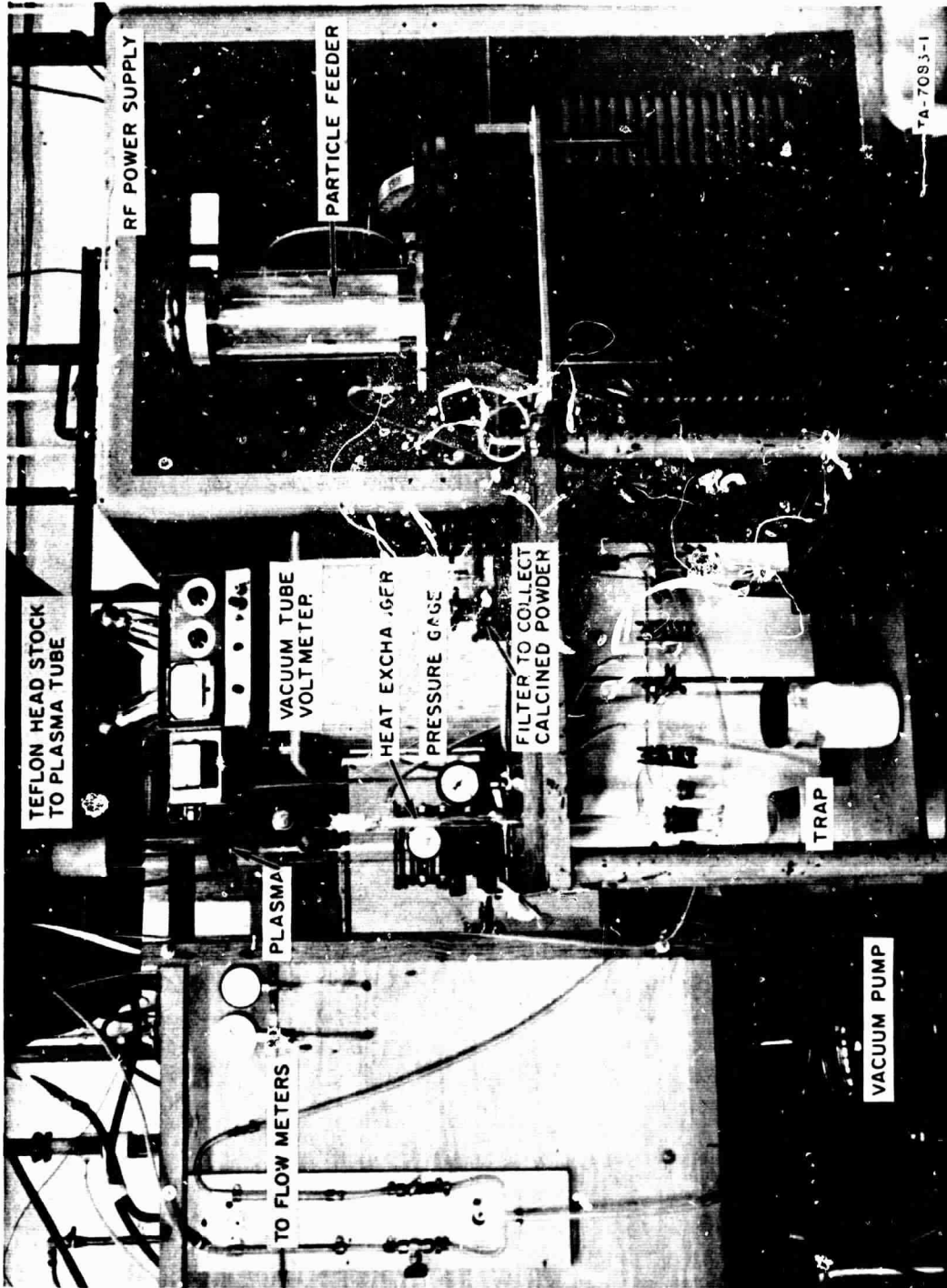FIG. 2 CALIBRATION CURVE FOR ZINC OXIDE PIGMENT FEED RATE

FIG. 3   PARTIAL VIEW OF PLASMA CALCINING APPARATUS

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Rotary action of the brush and thrashing against the trip bars broke up the pigment aggregates. Tests were made at several rotational speeds, and a speed of approximately 1700 rpm was found to yield a good dispersion. The speed of rotation was monitored stroboscopically. The powder disperser was coupled as closely to the plasma reactor as feasible in order to reduce the amount of flocculation while the particles were being transported to the plasma reactor. A photograph of the particle disperser is shown in Fig. 4. In this configuration the disperser and its air driven motor were mounted above the plasma reactor. The particles from the disperser entered the top of the reactor and passed through the plasma. The disperser was relocated when downstream injection of pigment particles was required.

3. Plasma Reactor

The plasma reactor was constructed from a 57-mm-diameter quartz tube. Gases passed downward through the vertical tube. The power coil for the induction plasma was outside this tube. The primary plasma zone was approximately 3 inches long. The quartz tube surrounding the plasma was cooled by a laminar flow of gas down the inside surface of the wall. This gas cooling sheath was created by the use of a concentric inner tube located above the plasma zone. Cool gas passed down the annulus between the inner and outer tube. The inner tube was flared at the bottom to provide for jetting the cooling gas sheath with sufficient velocity to project it along the hot wall. The shape and alignment of this inner tube is critical to the successful operation of this type of plasma reactor.

Feed gas (argon/oxygen mixture) carrying the pigment particles was injected downward. Examination of particle motion in the reactor indicated that gas flow through the primary plasma zone approximated plug-flow. With downstream injection, the particles were rarely transported above the injection port, and flow patterns in the reactor indicated that the plasma gases traveled through the reactor tube to the particle feed injection port in plug-flow, while below this point the particle feed gases and hot gases from the plasma were turbulently mixed. Thus
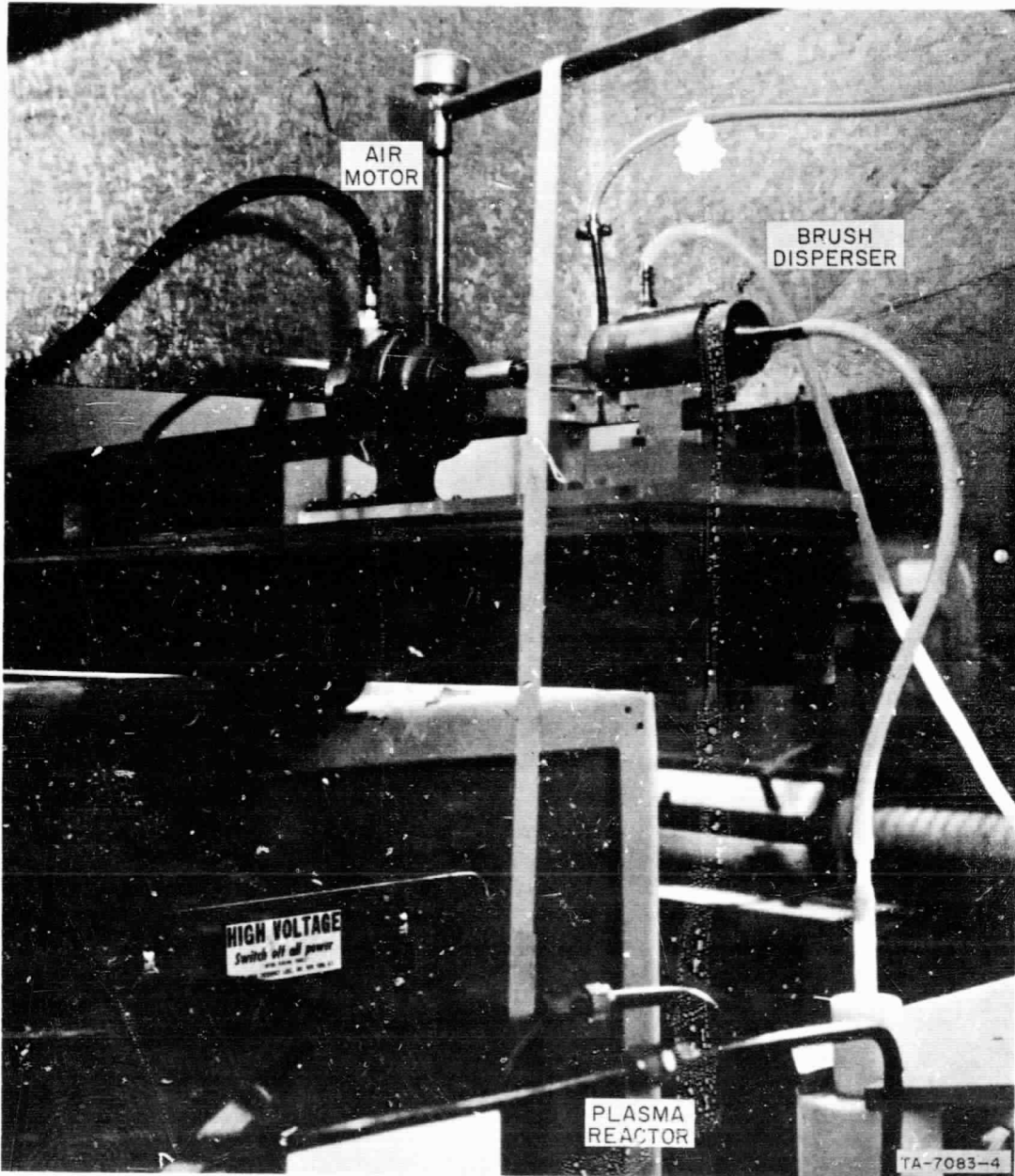
FIG. 4   ROTATING BRUSH PARTICLE DISPERSER

the lower part of the reactor appeared to behave more like an ideal back-mix reactor than a plug-flow reactor. For the downstream injection experiments, average residence time was calculated on the assumption that all of the reactor below the particle injection port behaved as an ideal back-mix reactor.

The bottom of the reactor was cooled by a water reservoir surrounding the plasma tube. As soon as the gases left the reactor side port, they were cooled further by heat exchangers. Additional energy was also lost by radiation from the plasma and by convection from the quartz reactor tube wall.

Power was supplied by a 50-kw Lepel induction power supply operating at 7.5 MHz. Low power settings were used, typically a plate current of 1.3 amp and a plate potential of 3000 volts. The largest factor in controlling the effective gas temperature was the point of particle injection and the amount of gas dilution used. Both experimental and calculated results indicated that particles injected through the plasma were being over-heated, and most of the runs were obtained with downstream injection. Gas leaving the plasma reactor was cooled by passing through straight-bore, water-cooled condensers (heat exchangers). Sufficient cooling was required to lower the gas temperature to a safe level for the filter paper. Most of the pigment put through the plasma reactor either remained in the bottom of the reactor tube or deposited on walls of the condensers and a trap at the bottom of the second condenser. A small proportion of the powder was collected on the filter.

A Millipore 142-mm filter holder was used for the final pigment collection. A flat circular filter paper with 0.45 μ pore size was used (Millipore type HA). This paper allows air passage of 13 feet^3/min at 10 psi differential pressure. Total flow rate in our experiments varied up to a maximum of approximately 4 feet^3/min.

Vacuum pumping the Millipore filter proved to be ineffective because of the low volumetric gas flow rates through the vacuum pump and the deleterious effect of this flow on the pump. Gas was forced through the filter by maintaining pressures higher than atmospheric pressure in the

plasma reactor. However, at the high volumetric flow rates required in some runs to obtain low average gas temperatures in the reactor, excessive pressures were required. Later the filter was coupled to an industrial vacuum cleaner with an air vent ahead of the Millipore filter. At high gas flow rates the excess gas was discharged through this vent, while at low flow rates additional coolant air was pulled in through the vent. Since the air vent was attached to a separate filtering system, no contamination of either the pigment in the Millipore filter at low plasma flow velocities or the atmosphere at high flow velocities was encountered.

IV   EXPERIMENTAL RESULTS

The first experiment involved the injection of silicated zinc oxide through the plasma. Argon flowing at 26 ℓ/min was used as the carrier gas. Although the pigment deposited within the reaction chamber was white, pigment deposited within the first heat exchanger was gray (see Fig. 5). X-ray diffraction analysis indicated that metallic zinc was present in considerable proportions in the gray powder. Evidently some of the zinc oxide disproportionates in the plasma to form zinc vapor, which passes out of the reaction chamber before condensing. The sharp temperature drop encountered at the walls of the first heat exchanger cause deposition of metallic zinc. Four additional unclassified short runs were made with oxygen additions to the argon feed. This eliminated the formation of metallic zinc, but the resulting zinc oxide had a yellow hue. With sufficient oxygen additions to prevent reduction of zinc oxide to metallic zinc, the plasma became unstable and easily extinguished. Consequently, these four runs were of short duration and an insufficient amount of material was produced.

A heat transfer analysis of particle heating in the plasma was made to guide further experiments. The reactions involved in the heat treatment of both silicated zinc oxide and zinc orthotitanate are not well understood, but they are believed to depend on the residence time at temperature as well as the temperature itself. The residence times in plasma heating are much shorter than residence times in kiln calcining. Furthermore, back-mixing occurs in the reactor with downstream injection. This means that some of the particles leave immediately, while others remain in the reactor for a long period. The probability that a particle remains in the reactor decays exponentially with time. Thus, guidelines for determining the temperature to which the pigment particles should be heated in a plasma are difficult to determine.
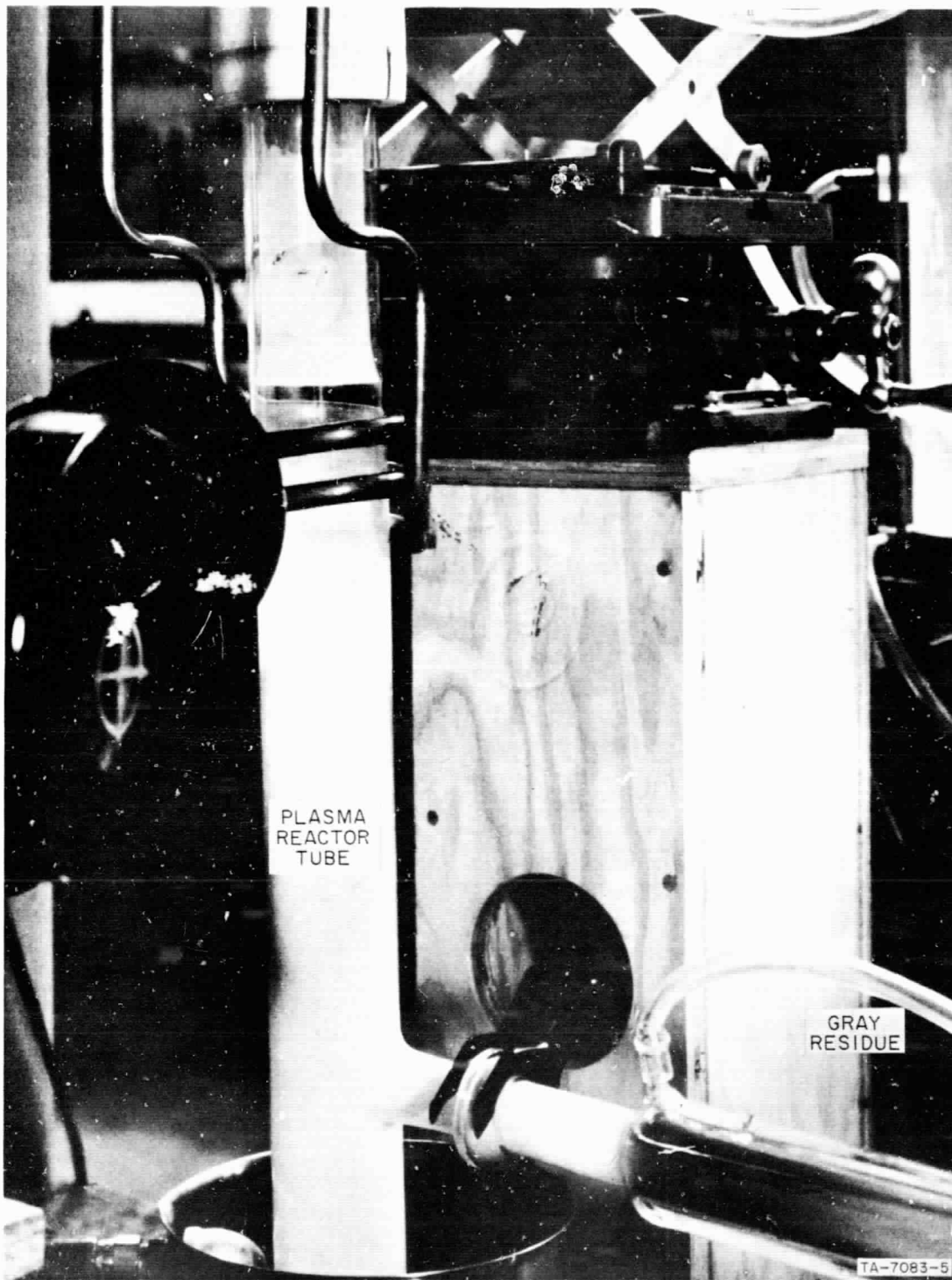
FIG. 5   METALLIC ZINC DEPOSITION (GRAY RESIDUE)
ON WALL OF HEAT EXCHANGER

Particle heating occurs by several mechanisms including thermal conduction through the boundary layer surrounding the particle, radiation, electron bombardment, and atom recombination on the particle surface. Since the plasma is optically thin, radiation contributes only a relatively insignificant heat flux. Electron bombardment and atom recombination can be viewed as mechanisms tending to increase the effective thermal conductivity of the plasma. Consequently, the process of particle heating can be analyzed on the basis that plasma boundary layer conduction is the dominant means of heat transfer to the particle surface. The degree of particle heating obtainable will be influenced by the temperature gradients across the boundary layer, the thermal and transport properties of both the plasma and the particles, and the geometric and flow parameters of the system.

The plasma temperature and plasma shape are not uniform. Because of the steep temperature gradients (and hence the steep enthalpy gradients and velocity gradients) that exist in the plasma, and because not all of the particles can be injected into the hottest part of the flame, an exact analytical solution of the plasma-to-particle heat transfer problem is extremely formidable. The usual practical approach has been to determine empirically the proper operating conditions for a well characterized material, and then to estimate the changes in operating conditions required for other materials on the basis of a "relative difficulty of heating" parameter derived for each material by means of a simplified approximate heat transfer analysis.

One such simplified analysis has been carried out at Stanford Research Institute by Engelke.[5,6] He considers a hypothetical model in which particle heating occurs in a plasma of finite length, over which the plasma enthalpy and velocity are uniform. Particles are considered to enter the flame with zero axial velocity and to be subsequently accelerated over the effective flame length by viscous drag. Boundary layer thermal conduction is taken to be the dominant heat transfer mechanism. Temperature gradients within the particles are ignored. Analysis of this model involves simultaneous solution of the drag and heat conduction equations, so that the time required for a particle to

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traverse the flame length can be equated to the time required for the particle to be heated to some specified temperature. After simplification, the final solution can be put in the form:

$$\left[\frac{S(\lambda\Delta T)^2}{V\mu}\right]_{plasma} \geq \left[\frac{L^2 D^2}{16\rho}\right]_{particles}$$

where S is the effective plasma flame length, λ is the mean boundary layer thermal conductivity, ΔT is the mean boundary layer temperature gradient, V is the mean plasma velocity, μ is the mean plasma viscosity, L is particle heat content per unit volume of the particle at the specified ultimate temperature relative to that of the solid at room temperature, D is the mean particle diameter, and ρ is the mean particle density.

Since the particle temperature always remains a small fraction of the plasma temperature, it follows that the mean thermal conductivity λ in the boundary layer and the temperature gradient ΔT across the boundary layer are each governed almost entirely by the enthalpy and composition of the plasma. Consequently, λ and ΔT have been lumped together with the plasma parameters on the left-hand side of the equation.

The Engelke equation provides a very approximate but useful guide for estimating the effect of plasma on the particles or for adjusting plasma conditions to obtain optimum particle heating. Although the equation was developed for use with larger particles encountered in plasma spraying, it appears to be equally suitable for analysis of fine pigment particles.

This equation was used to examine the heating of zinc oxide pigments in the plasma. The hot zone of the plasma was assumed to be 15 cm long, the particle diameter was taken as 1 μ, and the pigment heat content was assumed to be 6620 cal cm^{-3}. The flow rate was 1 $foot^3$/min (STP), which corresponds to 20 ℓ/min and is typical of the experimental conditions that were used. The parameter $\lambda\Delta T$ was estimated from Amdur and Mason's data[7] and is deemed to be conservative. The calculations are presented in Table II. The results of these calculations show that the heat input of the plasma, 55 cal cm^{-3} g^{-1}, is far in excess of the

heat input required to obtain the desired degree of particle heating, 5.1×10^{-3} cal cm^{-3} g^{-1}. In fact, the heating rate is about 4 orders of magnitude greater than that required to heat the particles. This large disparity results primarily because of the extremely high temperature of the plasma and the very fine particle size. These results suggest that it is not necessary to pass well-dispersed pigment particles through the plasma; rather it would be more desirable to inject particles downstream with sufficient ballast gas to attain a lower average gas temperature after mixing with hot gas from the plasma.

TABLE II

PLASMA HEATING CALCULATION FOR 1 μ ZnO PARTICLE

Reactor cross section area A	0.0124 foot^2
Plasma length S	15 cm
Plasma average flow velocity V (10,000°K) for 1 foot^3/mm (STP)	1.36×10^3 cm/sec
Heating parameter $\lambda\Delta T$ (estimated from Amdur and Mason's data[7])	4 cal cm^{-2} sec^{-1}
Plasma viscosity μ	3.2×10^{-3} g cm^{-1} sec^{-1}
ZnO sensible heat L	6720 cal cm^{-3}
Particle diameter D	1×10^{-4} cm
Particle density ρ	5.47 g cm^{-3}

$$\frac{S(\lambda\Delta T)^2}{V\mu} = 55 \text{ cal } cm^{-3} g^{-1}$$

$$\frac{L^2 D^2}{16\rho} = 5.15 \times 10^{-3} \text{ cal } cm^{-3} g^{-1}$$

A series of experiments were run with both silicated zinc oxide and zinc orthotitanate at different average gas temperatures. The average gas temperature was determined from the plate voltage and plate current of the Lepel power supply, the gas flow rate, and the heat capacity of argon, which was the primary gas present in the system. From previous

calibration experiments, it was known that the heat input to the plasma was approximately 50% of the plate power output of the power supply. On this basis

$$\dot{q} = 5.7(E_p)(I_p)$$

where $\dot{q}$ is the heating rate in calories per minute. The gas temperature in excess of ambient, ΔT, is

$$\Delta T = 25(E_p)(I_p)/J$$

where the total gas flow rate J is given in liters per minute (STP). Total flow rate includes both the flow rate of gas along the wall of the quartz tube reactor near the plasma (sheath flow) and flow gas from the particle feeder, (see Fig. 1). The procedure for each run was to calculate the necessary total flow rate for a specified average gas temperature for fixed values of plate voltage and plate current. During a run the plate voltage and plate current could be held constant provided that the gas flow rate and gas mixture were held constant. Consequently, it was possible to specify a total gas flow rate to give a specified ΔT, to record the voltage plate current and the actual flow rates through all the flow meters, and finally to calculate the real gas temperature for each run.

A variety of calibrated flow meters were used to achieve considerable variety in the gas flow rate and effective gas temperature. Oxygen content of the total gas mixture generally ranged between 5 and 10%. Higher oxygen content led to plasma instability, particularly if the oxygen was introduced into the sheath flow. The experiments were either 30 or 45 minutes in duration, and power output, flow rates, temperatures, and pressures were recorded periodically during the run. At high flow rates or when the pressure on the exit side of the flow meters was greater than 1 atm, pressures near the flow meters were recorded and corrections were made for the pressure effect on the meter flow rate charts. Once flow and plasma power conditions were established, a remarkably stable process resulted when pigment particles were injected downstream from the plasma. The results of experiments 2 through 11, covering the downstream injection experiments, are shown in Table III.

Three samples of pigment residue collected from each of runs 1 through 11 have been submitted to IIT Research Institute for evaluation.

Table III

PROCESS DATA ON PLASMA CALCINING EXPERIMENTS WITH DOWNSTREAM PIGMENT INJECTION

RUN	SAMPLE	RUN PERIOD, min	PARTICLE FEED RATE, g/min	OPERATING PRESSURE, psig	POWER		FLOW RATE, l/min					OXYGEN, %	ΔT, C	τ, sec	REMARKS
					E_p, volts	I_p, amps	Sheath Argon	Sheath Oxygen	Feeder Argon	Feeder Oxygen	Total				
2	ZnO #1	30	1	0	3200	1.7	11	0	4	1	16	6.25	8500	3.9	Intermittent powder flow.
3	ZnO-#1	30	1	0	3600	1.2	11.75	1.0	12.8	0	25.6	4.0	4200	2.4	Oxide yellow at inlet neck, flow steady.
4	ZnO-#1	33	1	1	3400	1.2	34.7	0	33.4	2.5	70.6	4.5	1445	0.88	
5	ZnO-#1	30	1	2.4	3500	1.2	44	0	136	5.0	185	2.8	570	0.33	Very turbulent in reactor.
6	ZnO-#1	49	1	0.2	3200	1.5	33.4	0	23.9	2.5	49.8	5.0	2410	1.25	Less turbulence than run 5, oxide yellow in reactor and inlet neck, but whitens as it cools.
7	Zn_2TiO_4-#2	45	1	0.9	3300	1.4	32.5	0	52.4	4.0	88.9	4.5	1300	0.70	
8	Zn_2TiO_4-#2	15	1	0	3300	1.6	27.2	0	21.4	2.5	51.1	4.9	2580	1.2	Oxide product gray in heat exchanger.
9	Zn_2TiO_4-#2	45	1	1.0	3400	1.4	58.5	0	33.4	6.45	98.3	6.6	1270	0.63	Oxide yellow at inlet neck, powder in heater changes, less gray than run 8.
10	Zn_2TiO_4-#2	45	1	0.75	3400	1.7	35.5	0	37.0	9.0	81.5	11	1770	0.76	Oxide yellow at inlet neck, other oxide is white.
11	Zn_2TiO_4-#2	45	1	1.0	3000	1.3	22.4	0	63	11.3	96.7	11.7	1010	0.64	

V   CONCLUSION AND RECOMMENDATIONS FOR FUTURE WORK

Plasma calcination of pigment particles is a stable and controllable process. There appear to be no technical obstacles to continuing runs for much longer periods than those employed in the experiments described here. Final conclusions on the value of plasma calcining for silicated zinc oxide and zinc orthotitanate pigments will depend upon evaluations to be performed by IIT Research Institute under NASA sponsorship.

The present program was very limited. Experiments at a variety of gas temperatures similar to those performed on silicated zinc oxides sample 1 and zinc orthotitanate sample 2 should be extended to the remaining 6 lots of pigment powders received from IIT Research Institute. Plasma calcining experiments of longer duration would provide more material for evaluation. The plasma-calcined pigment particles should be characterized by the research group doing the plasma calcining and these results should be compared with the feed powders. This characterization should include chemical analysis, X-ray diffraction, and analysis of the size and shape of the resulting pigment particles. Electron microscopy and Fisher particle size analysis are recommended techniques for size analysis of these powders.

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