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**CHAR PARTICLE FRAGMENTATION AND ITS EFFECT
ON UNBURNED CARBON DURING PULVERIZED COAL
COMBUSTION**

Quarterly Report for the Period

January 1, 1993 - March 31, 1993

Grant DE-FG22-92PC92528

Prepared for

THE UNITED STATES DEPARTMENT OF ENERGY

**James Hickerson
Project Officer
Pittsburgh Energy Technology Center
Pittsburgh, PA 15236**

Submitted by

Mr. Ruben Diaz and Professor Reginald E. Mitchell

April 1993

**HIGH TEMPERATURE GASDYNAMICS LABORATORY
Mechanical Engineering Department
Stanford University**

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April 1993

High Temperature Gasdynamics Laboratory
Department of Mechanical Engineering
Stanford University

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RESEARCH OBJECTIVES

This document is the second quarterly status report of work on a project concerned with the fragmentation of coal char particles that is being conducted at the High Temperature Gasdynamics Laboratory at Stanford University, Stanford, California. The project is intended to satisfy, in part, PETC's research efforts to understand the chemical and physical processes that govern coal combustion. The work is pertinent to the char oxidation phase of coal combustion and focuses on how the fragmentation of coal char particles affects overall mass loss rates and how char fragmentation phenomena influence coal conversion efficiency. The knowledge and information obtained will allow the development of engineering models that can be used to predict accurately char particle temperatures and total mass release rates. In particular, the work will provide insight into causes of unburned carbon in the ash of coal-fired utility boilers and furnaces. Work is to be performed over the three-year period from September 1992 to September 1995.

The proposed study has relevance to char particle fragmentation and its effect on mass loss rates during pulverized coal combustion. Depending on coal type, a significant number of char particles are formed during devolatilization that are categorized as being cenospheres or mesospheres - particles that have relatively large void volumes within them. Large voids at the outer surfaces of particles allow oxygen to consume the inner particle material. As a consequence, particles may fragment. Fragments burn at rates governed by their individual sizes and not at rates determined by the sizes of their parent char particles. Thus, the overall mass loss rates of char particles that fragment extensively can not be predicted accurately without accounting for the effects of fragmentation. In this study, to eliminate the complications associated with the complex

composition of coals, combustion tests are performed using synthetic chars having particle morphologies similar to those of the char particles formed during coal devolatilization.

The overall objectives of the project are: (i) to correlate char particle porosity with fragmentation phenomena, (ii) to determine if mineral matter in the coal affects fragmentation patterns, and (iii) to relate the effects of fragmentation events to unburned carbon in ash. The knowledge obtained during the course of this project will be used to predict accurately the overall mass loss rates of coals based on the mineral content and porosity of their chars. The work will provide a means of assessing reasons for unburned carbon in the ash of coal fired boilers and furnaces.

The project is divided into four research tasks. Specific objectives associated with each task are as follows:

Task 1: Production and Characterization of Synthetic Chars

Objective: The objective of this task is to produce and characterize synthetic chars with controlled macroporosity and known mineral content. Densities, porosities, pore size distributions, and total surface areas will be measured. Chemical analyses will be performed to determine the composition of chars that have been laden with pyrites, calcites, silica, and gypsum.

Deliverables: Results of this task will yield well-characterized materials for use in combustion and fragmentation studies associated with Tasks 2 and 3 of this project. Particles in the size ranges 75 - 90 μm , 90 - 106 μm and 106 - 125 μm that have porosities up to 75% will be produced.

Task 2: Baseline Char Combustion Experiments

Objectives: The objectives of this task are to design and fabricate an entrained flow reactor and a solids extraction probe and to determine gaseous conditions for diffusion-limited combustion of the synthetic chars. The extent to which particles fragment during the extraction process will be characterized.

An additional objective is to employ thermogravimetric analysis to determine the extent to which the overall particle burning rates of the mineral-laden synthetic chars are catalyzed in the gaseous environments that will be used in the fragmentation studies.

Deliverables: The following will result after completion of this task:

- An entrained flow reactor capable of simulating environments typical of pulverized coal combustors and a solids extraction probe that permits sampling of partially reacted chars at different residence times in the reactor.
- Characterization of the extent to which particles fragment during the extraction process.
- Oxygen concentrations and gas temperatures that yield diffusion-limited burning of the synthetic chars produced.
- Characterization of the extent of catalysis in the gaseous environments employed due to mineral constituents of the synthetic chars.

Task 3: Char Fragmentation Studies

Objective: The overall objective of this task is to obtain the data necessary to understand how the porosity of char particles affects their fragmentation behavior and how the minerals in char particles influence their fragmentation patterns.

Deliverables: The following will result after completion of this task:

- A measure of fragmentation events that result as a consequence of burning at diffusion-limited rates in various gaseous environments as a function of particle porosity.
- A measure of how the type of mineral and the mineral content of char affects its fragmentation patterns.

Task 4: Fragmentation Modeling

Objective: The objective of this task is to develop and validate a fragmentation model that can be incorporated into a char oxidation model.

Deliverable: The successful completion of this task will yield a char fragmentation model that describes the results of Task 3 experiments. The model will be capable of accurately predicting significant fragmentation events in gaseous environments typical of pulverized coal combustors. When combined with a char oxidation model, the extent to which fragments might extinguish and hence, contribute to unburned carbon in ash can be predicted.

TABLE OF CONTENTS

RESEARCH OBJECTIVES.....	i
TECHNICAL PROGRESS DURING THIS QUARTER.....	1
Summary.....	1
Task 1. Production and Characterization of Synthetic Chars.....	2
Char Preparation.....	2
Char Characterization.....	5
Task 2. Baseline Char Combustion Experiments.....	15
Laminar Flow Reactor and Sampling Probe Assembly.....	15
Char Oxidation Experiments in the PTGA.....	15
PLANS FOR NEXT QUARTER.....	16
REFERENCES.....	16

TECHNICAL PROGRESS DURING THIS QUARTER

SUMMARY

The information reported is for the period January 1 to March 31, 1993. During this quarter, activities were undertaken in Tasks 1 and 2. Additional samples of synthetic char were produced and characterized. These chars were prepared with lycopodium to polymer mass ratios of 0:1, 1:2, and 1:8. Combined with the char prepared with a lycopodium to polymer of 1:4 that was discussed last quarter, we now have synthetic chars varying in porosity from 17% to 37%. These chars will be used in the forthcoming char fragmentation experiments.

Using both a tap density technique and mercury intrusion porosimetry on synthetic char with no macropores, a value of 0.39 was determined for the bed void fraction of tightly packed particles in the 75-125 μm size range. The true density of the synthetic char was found to be 1.58 g/cc using helium pycnometry. From these measurements, the apparent density and porosity for each batch of char were determined. A linear relationship was observed between the particle porosity and the wt-% lycopodium used in the synthesis. Using this relationship, synthetic chars of specified porosity can be produced. SEM pictures were taken of the synthetic char and these photographs showed particles with pores of fairly uniform size (nominally 20 μm in diameter).

The mechanical grinding procedure was refined to obtain a greater size fraction of particles in the 75-125 μm size range of interest. An investigation of the log-normal distribution function for describing the measured particle size distribution was performed. It was noted that a log-normal distribution function adequately described the breadth and mean size of the size classified particles but does not completely capture the wings of the distribution. The log-normal distribution will be useful, however, in describing the changing distribution that results from mass loss and fragmentation, phenomena that tend to shift the distribution to smaller sizes.

Preliminary experiments were performed with the pressurized thermogravimetric analyzer (PTGA). Combustion in air of synthetic chars having 17% and 24% porosity revealed that the overall particle burning rates of the chars were essentially the same. In the combustion tests, temperature was ramped from 300 K to 1173 K at 25°C/min. This indicates that under these conditions there were no diffusional resistances to particle burning - the

overall particle burning rates were controlled by the chemical reactivity of the particle material. The preliminary experiments also indicate that there is no measurable effect of any residual lycopodium that may remain in the char. The char having 17% porosity was lycopodium-free.

Also during this quarter, a Perkin-Elmer elemental analyzer (on loan from Sandia National Laboratories) was set-up and prepared for testing. This instrument will be used for proximate analyses. A support structure for the reactor traverse system and the flow control instrumentation were also built. The traverse will allow measurements to be obtained at different residence times as well as permit off-axis locations within the reactor to be sampled. In addition, design of the solids sampling probe was initiated. The probe will permit quenched samples of partially reacted chars to be obtained.

TASK 1: PRODUCTION AND CHARACTERIZATION OF SYNTHETIC CHARS

Char Preparation

Using the procedure outlined in the previous quarterly status report, three additional batches of synthetic char were produced with different lycopodium to polymer mass ratios. Varying this ratio varies the macroporosity of the chars produced. The lycopodium to polymer mass ratios used in the synthesis process were 0:1 (no lycopodium added), 1:8 (one part lycopodium to 8 parts polymer), and 1:2 (one part lycopodium to 2 parts polymer). The carbon black to polymer mass ratio was held constant at 1:3 for each of these synthetic chars. The carbon black affects the microporosity of the particles produced. These synthetic chars were used to determine a relationship between the porosity of the particle and the amount of lycopodium added.

Size Classification

During the quarter, the mechanical grinding procedure was refined to obtain a greater fraction of particles in the size range 75-125 μm . Mechanical grinding is performed with a Spex Industries, Inc. shatterbox, where a metal puck is rotated within a sealed metal container. During forced rotation, the puck bounces off the inner walls of the container pulverizing the char particles placed between the puck and walls. Only a few seconds is needed to pulverize about 20 grams of material.

Originally, a tungsten carbide puck and container were used to pulverize the char particles. Due to the material's hardness, the particles were efficiently pulverized to particles

with diameters under 75 μm . A different puck and container combination made of hardened steel, a material that is not as hard as tungsten carbide, produced a greater percentage of particles over 75 μm using the same pulverization time. After several trials with the hardened steel puck and container, the pulverization time that gave the greatest percentage of particles having sizes within the 75-125 μm size range was found to be 10 seconds. The pulverization procedure now consists of the following steps: (1) mechanical grinding for 10 seconds; (2) sieving; (3) further mechanical grinding of material greater than 125 μm ; and (4) resieving. The sieved material is then subjected to wet sedimentation to remove fines. The size classified material is then stored in a desiccator for use in future fragmentation experiments.

In the previous quarterly report we showed that a gaussian distribution function depicted the measured wt-% distribution of the size classified particles quite well. The mean diameter and the wings of all three size ranges were accurately described. An alternative distribution function that has wide use in particle statistics is the log-normal distribution. It accurately describes particle size distributions that are skewed toward the smaller size ranges (Aitchison and Brown, 1957). This distribution is given by:

$$l(x) = \frac{100}{\sqrt{2\pi} x \ln \sigma_g} \exp \left[- \left(\frac{\ln(x / x_l)}{\sqrt{2} \ln \sigma_g} \right)^2 \right]$$

where x is the particle diameter, x_l is the median diameter, and σ_g is the geometric standard deviation, (a measure of the breadth of the distribution). With this distribution, approximately 68% of the particles have diameters between $\sigma_g x_l$ and x_l / σ_g . A value of σ_g greater than 2 implies that the sample has a very broad distribution. As the value of σ_g approaches one, the sample becomes monodisperse (Ghosal *et al.*, 1993).

Employing a nonlinear programming solver that uses sequential quadratic programming, size distributions of sieved particles obtained with a Coulter Multisizer were fit to log-normal functions. The results of the fits are shown in Figs. 1-3. As noted, the log-normal distribution function adequately describes the breadth and mean diameter of all three particle distributions but does not completely capture the wings. Although this log-normal distribution does not describe the size distribution for the sieved particles as well as the gaussian distribution, we believe that it will be more accurate as the size range tends to broaden preferentially toward smaller sizes because of particle burning and fragmentation. A

gaussian distribution function would predict broadening symmetric about the mean particle size, a phenomenon that is not expected.

Figure 1. Weight Percent of particles having diameters between x and $x+dx$ versus diameter for particles in the 75 - 90 μm size range. The solid line represents a log-normal distribution fit to the data.

Figure 2. Weight Percent of particles having diameters between x and $x+dx$ versus diameter for particles in the 90 - 106 μm size range. The solid line represents a log-normal distribution fit to the data.

Figure 3. Weight Percent of particles having diameters between x and $x+dx$ versus diameter for particles in the 106 - 125 μm size range. The solid line represents a log-normal distribution fit to the data.

Char Characterization

Char Apparent Density

The previous quarterly report described two methods used to determine the apparent density of the char particles: a tap density technique and mercury intrusion porosimetry. In the tap density technique, the apparent density is calculated from the measured bulk density of a tightly packed bed of particles and the bed void fraction. The bed void fraction for the synthetic char particles in the nominal size range of 75 - 125 μm was determined from a combination of the tap density technique and mercury intrusion porosimetry on char produced with only micropore-forming carbon black added. As explained in the previous report, there is uncertainty in the determination of the apparent density from porosimetry data because the nominal pore size of 20 μm (produced from the evaporation of the lycopodium spore) falls within the size range of interparticle voids. By excluding the macropore-forming lycopodium during the synthesis process, particles are produced for which the intraparticle pores and the interparticle spacings do not overlap in size. The separation in size of these two types of voids is evident in Fig. 4, which is a plot of the mercury porosimetry data for the macropore-free char. In the plot, both the incremental mercury intrusion volume and the cumulative volume are plotted versus the pore diameter, which decreases from left to right. As the mercury is progressively forced into smaller open spaces, there is a point (about 3 μm),

Figure 4. Mercury intrusion porosimetry data for synthetic chars produced with no macropore-forming lycopodium added and used to determine actual apparent density of the particles. The inset box is an expanded view of the incremental volume between 0.1 and 0.003 μm

where the incremental mercury intrusion volume drops to zero and the cumulative volume levels off. The incremental and cumulative volumes do not again increase until a void size of under 0.05 μm is reached (see inset for expanded view of the submicron region). This indicates that the interparticle voids are greater than 3 μm and that the intraparticle pores are only as large as 0.05 μm for the synthetic char without lycopodium. Using 3 μm as the "cut-off" diameter (openings larger than this constitute interparticle voids, smaller than this intraparticle voids), the apparent density of the particles is found to be 1.33 g/cc. This is taken as the actual apparent density of this char, which contains only micropores. Using the tap density method on samples of this char, the bulk density was found to be 0.80 g/cc. Using these values of apparent and bulk densities, the bed void fraction was found to be 0.39.

To assess the applicability of this value of the bed void fraction to chars with macropores, the tap density technique and mercury intrusion porosimetry were performed on chars with lycopodium to polymer ratios of 1:8, 1:4, and 1:2. Figure 5 shows incremental and cumulative volumes for the char having a 1:2 ratio. The cumulative volume continuously increases, suggesting no distinct separation between interparticle and intraparticle voids. For this char, the apparent density determined via mercury porosimetry could potentially be in error.

Figure 5. Mercury intrusion porosimetry data for synthetic chars produced with a lycopodium to polymer ratio of 1:2. The cumulative volume continually rises throughout the above range of pore diameters.

The bulk densities of these chars were determined from the data shown in Fig. 6, which shows the mass of particles needed to fill various volumes. The slope of each line is the bulk density of the char. The plot shows that the bulk density of the char decreases as the relative amount of lycopodium increases.

Figure 6. The mass of synthetic char that packs various tube volumes for four different char samples, having various lycopodium to polymer ratios, in the particle size range 75-125 μm . A ratio of 0:1 corresponds to no lycopodium added. Lines represent least-squares fit to the data.

Values determined for bulk densities of the chars having different amounts of lycopodium added during the synthesis process are listed in Table 1. Also shown in the table are values calculated for apparent densities using 0.39 for the bed void fraction and values obtained from the mercury intrusion porosimetry data using a cut-off diameter of 21 μm . Since the spore of the lycopodium plant yields macropores that are nominally 20 μm , use of the 21 μm cut-off diameter appears to be reasonable. There is excellent agreement in the apparent densities determined with the two methods. The differences suggest an error of less than 5% in the apparent density determination via the tap density technique. As the porosity of particles increases, coalescence of pores increases and openings at the particle's outer surface can exceed 20 μm . For these particles, mercury intrusion porosimetry will not yield accurate values for apparent density. The tap density method for apparent density employing a bed void fraction of 0.39 will have to be used.

Table 1. Bulk and apparent densities, determined from tap density technique and mercury intrusion porosimetry, for various lycopodium to polymer mass ratios. (*Data used to determine bed void fraction).

lycopodium to polymer mass ratio	bulk density (from tap density technique) g/cc	apparent density (from tap density technique) g/cc	apparent density (from mercury porosimetry) g/cc
0:1	0.80	1.33	1.33*
1:8	0.73	1.20	1.14
1:4	0.69	1.13	1.14
1:2	0.61	1.00	0.99

Particle Porosity

The particle porosity is calculated from the apparent and true densities as follows:

$$\theta = 1 - \rho_a / \rho_t$$

where θ is the porosity, ρ_a is the apparent density and ρ_t is the true density. The true density is determined from helium pycnometry, helium being capable of penetrating the smallest pores within the particle and hence, filling all the intraparticle voids. The true density of the 1:8 char was determined to be 1.58 g/cc. This value for true density is taken to be the same for all the char samples. This is supported by the fact that the skeletal densities

of the different chars (as determined by mercury intrusion porosimetry) were found to be essentially the same, varying only 1% about a mean value of 1.42 g/cc. The skeletal density only approximates the true density since mercury can not penetrate the smallest pores within the particle. Incidentally, the small variation in skeletal density indicates the repeatability of the production of the chars.

With this true density and the apparent densities of the various char samples, values were calculated for particle porosity. The results are shown in Fig. 7, where porosity is plotted against weight percent lycopodium, where weight percent lycopodium is defined as the mass of lycopodium divided by the sum of the masses of lycopodium and polymer. In all cases, carbon black was added at a carbon black to polymer mass ratio of 1:3. In Fig. 7, it is seen that for 0 wt-% lycopodium, the particle porosity is about 17%. This porosity is produced from the addition of carbon black that creates micropores of about 0.05 μm and under. As the relative amount of lycopodium is increased, the particle porosity increases linearly in lycopodium weight percent range of 0 to 33%. At 33 wt-% lycopodium, the particle porosity is about 37%. During the next quarter the range of controllable porosity will be extended. It is hoped that the relationship between weight percent lycopodium and char particle porosity will remain linear up to the targeted upper value of 75%.

Figure 7. The porosity of the synthetic chars versus the weight percent of lycopodium. Carbon black was added to all chars at a mass ratio of carbon black to polymer of 1:3.

Initial batches of char with 85 wt-% lycopodium (which corresponds to about 67% porosity assuming a linear relationship), were produced and size-classified. A negligible amount of particles were obtained in the 75-125 μm size range. We hypothesize that the higher porosity reduces the structural strength of the particles and with mechanical grinding, the particles are reduced to sizes well below the desired range. For these higher porosity particles, hand grinding with mortar and pestle exclusively, may replace mechanical grinding.

Scanning Electron Micrographs of Synthetic Chars

Figures 8-15 are scanning electron micrographs (SEM) taken of synthetic chars having the following measured levels of porosity: 17%, 24%, 28%, and 37%. Each micrograph displays a bold, white, reference line whose length corresponds to 100 μm . For each value of the porosity there is a set of two micrographs. The first in each set shows several particles whose sizes are fairly uniform and that generally fall within the desired nominal size range of 75-125 μm . The second micrograph in each set shows a close-up of a typical particle in the first micrograph.

In Figs. 8 and 9, which show particles having an average porosity of 17%, the large pores seen in the other figures are not present in these particles. As mentioned earlier, the pore sizes of this char (made with only carbon black added and no lycopodium) are no larger than 0.05 μm , and are not visible in this image. In the other figures, the large, approximately circular, voids in the particles are the nominally 20 μm pores produced from the evaporation of the lycopodium spores. As noted, the number of these pores in each particle increases with the average porosity of the pores. In Fig. 15, where the particle porosity is about 37%, a pore of about 32 μm is noted (the pore near the center of the picture). This pore is most likely the product of the coalescence of two pores formed from evaporation of the lycopodium spores. As the lycopodium weight percent is increased, the likelihood of pores coalescing is increased. Hence, we expect that particles having porosities greater than about 37% will have a wider distribution of macropore sizes. Also seen in Fig. 15, are small circular nodules in each of the pores. Since these nodules are observed only in the regions where spores have evaporated, the nodules could be residue from lycopodium pyrolysis. We plan heat treatment experiments in inert environments to determine if these nodules can be removed.

Chemical Characterization

In addition to the above work in determining the particle apparent density and porosity, the elemental analyzer, on loan from Sandia National Laboratories, arrived and was set up. It will be used to perform proximate analysis on the synthetic chars.

Figure 8: 112X magnification SEM photograph of synthetic char particles sized within a range of 75-125 μm and produced with an average porosity of 17%. The bold white line represents 100 μm .

Figure 9: 552X magnification SEM photograph of a synthetic char particle sized within a range of 75-125 μm and produced with an average porosity of 17%. The bold white line represents 100 μm .

Figure 10: 112X magnification SEM photograph of synthetic char particles sized within a range of 75-125 μm and produced with an average porosity of 24%. The bold white line represents 100 μm .

Figure 11: 544X magnification SEM photograph of a synthetic char particle sized within a range of 75-125 μm and produced with an average porosity of 24%. The bold white line represents 100 μm .

Figure 12: 124X magnification SEM photograph of synthetic char particles sized within a range of 75-125 μm and produced with an average porosity of 28%. The bold white line represents 100 μm .

Figure 13: 604X magnification SEM photograph of a synthetic char particle sized within a range of 75-125 μm and produced with an average porosity of 28%. The bold white line represents 100 μm .

Figure 14: 120X magnification SEM photograph of synthetic char particles sized within a range of 75-125 μm and produced with an average porosity of 37%. The bold white line represents 100 μm .

Figure 15: 604X magnification SEM photograph of a synthetic char particle sized within a range of 75-125 μm and produced with an average porosity of 37%. The bold white line represents 100 μm .

TASK 2: BASELINE CHAR COMBUSTION EXPERIMENTS

Laminar Flow Reactor and Sampling Probe Assembly

The support structure for the laminar flow reactor has been built and the traverse mechanism has been purchased. The three-axes traverse system will allow measurements to be obtained at different residence times as well as allow off-axis locations within the reactor to be sampled.

A preliminary design of the solids sampling probe was completed. The probe consists of four concentric stainless-steel tubes with an outer diameter of 1.9 cm. The outer annuli are for cooling water; the annulus surrounding the center tube is for the helium quench gas. The hot reactor gases, char particles, and helium will be removed from the reactor through the center tube. Particles will be collected on a filter; the gases (N_2 , O_2 , H_2O , CO_2 , and He) will be exhausted to the environment. The probe and support assembly will permit samples of partially reacted chars to be extracted from the flow reactor at selected residence times. The final design and fabrication of the probe will be completed during the next quarter. Design of the particle collection system will be initiated.

Char Oxidation Experiments in the PTGA

Oxidation experiments were performed in the pressurized thermogravimetric analyzer (PTGA) with samples of the porous synthetic chars produced. Chars with porosities of 17% and 24% were heated to 1173 K at 25°C/min within an air environment at atmospheric pressure. The weight loss curves for the two samples have approximately the same maximum slope (~0.0017 g/min) and show the onset of weight loss occurring at approximately the same temperature (~673 K). Since the two weight loss curves are similar for chars with different porosities, this suggests that in the environment employed, the overall particle burning rates were limited by the intrinsic chemical reactivity of the particle material and not by any mass transport effects. Since the char having a porosity of 17% contained no lycopodium, these preliminary experiments suggests that there is no measurable effect of the lycopodium residue (observed in the micrographs) on the reactivity of the char.

PLANS FOR NEXT QUARTER

- Synthetic char having lycopodium weight percents approaching 99%, (which corresponds to 75% porosity assuming a linear relationship), will be produced.
- Shakedown tests will be performed on the elemental analyzer using benzoic acid (for C/H compounds) and acetanilide (for C/H/N compounds).
- The solids sampling probe will be finished and tested. The particle feeder, reactant gas flow system, and the probe cooling and quench systems, will be assembled and tested.

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