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FINAL REPORT

JULY 1, 1989 - JUNE 30, 1990

Grant #G1194119
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ABSTRACT

Allotment Grant G1194119
Final Report for July 1, 1989 - June 30, 1990

This Final Report describes the activities of the Iowa State Mining and Mineral Resources Research Institute (ISMMRRI) at Iowa State University for the period July 1, 1989, to June 30, 1990. Activities funded by the Bureau of Mines, U. S. Department of the Interior, under Allotment Grant #G1194119, include research in mining- and mineral-related areas, education and training of scientists and engineers in these fields, administration of the Institute, and cooperative interactions with industry, government agencies, and other research centers.

Through ISMMRRI, significant research has been conducted in mining- and mineral-related disciplines, fulfilling the responsibility charged to it by Public Law 100-483 for Mining and Mineral Resources Research Institutes. During this period, ISMMRRI has supported research efforts to:

- Investigate methods of leaching zinc from sphalerite-containing ores.
- Study the geochemistry and geology of an Archean gold deposit and of a gold-telluride deposit.
- Enhance low-quality aggregates for use in construction.
- Pre-clean coal by triboelectric charging in a fluidized bed.
- Characterize the crystal/grain alignment during processing of yttrium-barium-copper-perovskite (1-2-3) superconductors.
- Study the fluid inclusion properties of a fluorite district.
- Study the impacts of surface mining on community planning.
- Assess the hydrophobicity of coal and pyrite for beneficiation.
- Investigate the use of photoacoustic absorption spectroscopy for monitoring unburnt carbon in the exhaust gas from coal-fired boilers.

The education and training program continued within the interdepartmental graduate minor in mineral resources includes courses in such areas as mining methods, mineral processing, industrial minerals, extractive metallurgy, coal science and technology, and reclamation of mined land. In addition, ISMMRRI hosted the 3rd International Conference on Processing and Utilization of High-Sulfur Coals in Ames, Iowa.

The Institute continues to interact with industry in order to foster increased cooperation between academia and the mining and mineral community. Keeping this objective in mind, ISMMRRI has several collaborative research projects in mineral processing, fuel science, and mined-land reclamation.

SECTION I

DESCRIPTION OF THE INSTITUTE

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DESCRIPTION OF THE INSTITUTE

The Iowa State Mining and Mineral Resources Research Institute (ISMMRRI) is actively fulfilling the objectives of the nationwide Mineral Institute program through its interdisciplinary research and education programs. Substantial research is conducted by Iowa State University faculty, students, and ISMMRRI staff in the areas of mineral characterization, mineral extraction, mineral processing, fuel science, and mined-land reclamation. The Institute is charged with promoting, implementing, and monitoring research and education programs in mining- and mineral-related fields, and providing information and support services to academia, the general public, government agencies, industry, and business.

The activities of the Iowa State Mining and Mineral Resources Research Institute are supported with funds provided by the Bureau of Mines, U. S. Department of the Interior, under Section 2 of Public Law 100-483, the State Mining and Mineral Resources Research Institute Bill of 1988. In fiscal year 1990, an allotment of $138,000 was provided under Section 1. These funds were supplemented with twice this amount by matching non-federal funds. The allotment funding is used to stimulate and support research in the basic and applied aspects of the mineral sciences at Iowa State University. Research projects supported this fiscal year are described in Section II of this Annual Report.

A minor in Mineral Resources, developed by ISMMRRI and the Graduate College, aims to familiarize graduate engineering and science students with disciplines, other than their own, within the areas of mining and mineral resources. The Mineral Resources minor and other educational programs sponsored by ISMMRRI are described in Section III of this report.

Executive Committee

The Executive Committee is responsible for developing Institute priorities and establishing an overall research program to carry out these priorities. In FY 1989, the committee was composed of the Director of the Institute for Physical Research and Technology, Michael M. Crow; the Vice Provost for Research at ISU, Pat Swan; and the Director of the Iowa State Mining and Mineral Resources Research Institute, Richard Markuszewski.

Mineral Resources Advisory Committee

The Mineral Resources Advisory Committee is composed of faculty members from several of the departments participating in the Institute's research and educational programs. The research expertise and interests of the Advisory Committee members represent a broad range of topics covered by the graduate research projects and the industrial collaborative efforts of the Institute. Specifically, this committee:

1. Selects and evaluates the courses taught specifically for and in support of the Mineral Resources minor;

2. Evaluates proposals submitted for ISMMRRI assistantships by potential graduate and faculty researchers in mining- and mineral resource-related projects;

3. Advises the Institute Director on the research projects funded by the Allotment Grant;

4. Makes recommendations to the Director on use of funds for broadening the scope of Institute programs.

Members of the Mineral Resources Advisory Committee

Robert C. Brown	Assoc. Professor of Mechanical Engineering
Norman L. Dietrich	Assoc. Professor of Landscape Architecture
Stanley J. Henning	Assoc. Professor of Agronomy
Dah-Yinn Lee	Professor of Civil Engineering and Committee Chairman
Richard Markuszewski	Director of Institute (ex-officio member)
Geraldine M. Montag[1]	Professor of Industrial Engineering
Ram Pandit[2]	Asst. Professor of Indust. & Mfg. Systems Engr.
John W. Patterson	Professor of Materials Science and Engineering
John M. Pitt	Assoc. Professor of Civil & Construction Engineering
Paul G. Spry	Assoc. Professor of Geological & Atmospheric Sciences
Thomas D. Wheelock	Professor of Chemical Engineering

[1] Term ended May 15, 1990.
[2] Term began May 15, 1990.

Institute Director

The Institute Director and his staff have the responsibility to operate the Institute and act as executive secretary to the Executive Committee. Funds allocated to the Institute are under the budgetary control of the Director with approval of the Executive Committee. The Director is advised by the Assistant Director, is aided by the Program Assistant, and interacts with the Mineral Resources Advisory Committee in an ex-officio capacity.

SECTION II

ACTIVE MINERAL INSTITUTE PROJECTS

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ACTIVE MINERAL INSTITUTE PROJECTS

Research Program

The Iowa State Mining and Mineral Resources Research Institute is committed to performing basic and applied research related to Iowa's mineral resources and for the general benefit of the nation's mineral industry. The Institute channels the majority of its Allotment Grant funds into supporting graduate student research under faculty guidance at Iowa State University. Qualified graduate students working on a mining- or mineral-related project are typically awarded research assistantships and may also receive support for research supplies, travel, analytical services, etc. Undergraduate research is not usually supported by the Institute, except in cases when an undergraduate hourly research assistant is needed for an ISMMRRI-supported project, or when the research is of an exceptional nature.

Student Projects

Listed below are student research projects supported by the Iowa State Mining and Mineral Resources Research Institute Allotment Grant for 1989-1990. The projects are classified into categories provided by the U. S. Bureau of Mines. A detailed description of each project is given in the following pages.

MINERAL EXPLORATION

Title:	A REGIONAL FLUID INCLUSION STUDY OF THE CAVE-IN-ROCK FLUORITE DISTRICT, ILLINOIS
Researchers:	Greg Fuhrmann and Dr. Paul G. Spry
Department:	Geological and Atmospheric Sciences
Title:	GEOCHEMICAL MODELING OF THE ARCHEAN LODE GOLD DEPOSIT AT JARDINE-CREVASSE MOUNTAIN, MONTANA
Researchers:	Ping Liu and Dr. Paul G. Spry
Department:	Geological and Atmospheric Sciences
Title:	GEOCHEMICAL STUDY ON THE GIES GOLD-TELLURIDE DEPOSIT, JUDITH MOUNTAINS, MONTANA
Researchers:	Xiaomao Zhang and Dr. Paul G. Spry
Department:	Geological and Atmospheric Sciences

EXTRACTIVE METALLURGY

Title:	LEACHING OF SPHALERITE-CONTAINING ORES
Researchers:	Brian Buss, Joseph E. Kincs, Dr. Thomas Wheelock, and Dr. Richard Markuszewski
Department:	Chemical Engineering and ISMMRRI

MINERAL PROCESSING

Title: HEAT OF IMMERSION OF COAL AND THREE-PHASE CONTACT ANGLE IN A COAL-WATER-OIL SYSTEM
Researchers: Xiaoping Qiu and Dr. Thomas D. Wheelock
Department: Chemical Engineering

Title: COAL BENEFICIATION UTILIZING TRIBOELECTRIC CHARGING IN A FAST FLUIDIZED BED
Researchers: David R. Tucholski and Dr. Gerald M. Colver
Department: Mechanical Engineering

Title: CRYSTAL/GRAIN ALIGNMENT DURING PROCESSING OF YTTRIUM-BARIUM-COPPER PEROVSKITE (1-2-3) SUPERCONDUCTORS
Researchers: Tim Green and Dr. Mufit Akinc
Department: Materials Science and Engineering

Title: ENHANCEMENT OF LOW-QUALITY LIMESTONE AND DOLOMITIC AGGREGATES FOR USE IN CONSTRUCTION
Researchers: John Vu and Dr. John Pitt
Department: Civil and Construction Engineering

MINED-LAND RECLAMATION

Title: THE IMPACT OF SURFACE MINING ON THE FT. DODGE, IOWA, AREA
Researchers: Lynne Spoden and Prof. Norman Dietrich
Department: Landscape Architecture

FUEL SCIENCE

Title: ON-LINE DETERMINATION OF UNBURNED CARBON IN AIRBORNE FLY ASH
Researchers: Jeff Dykstra and Dr. Robert Brown
Department: Mechanical Engineering

MINERAL CHARACTERIZATION

Title: CALORIMETRIC CHARACTERIZATION OF FLY ASH FOR MINING AND ENGINEERING APPLICATIONS
Researchers: Argha Saha and Dr. John Pitt
Department: Civil and Construction Engineering

New Student Projects for 1990-1991

The following graduate students, who have not been ISMMRRI students before, or who are pursuing an additional degree, have been selected by the Mineral Resources Advisory Committee to receive research assistantships for the 1990-1991 academic year. The students have agreed to minor in Mineral Resources and to perform research with their major professors on the mining and mineral-related topics given below.

Title INDUCTION TIME CHARACTERIZATION OF THE RELATIVE HYDROPHOBICITY OF COAL AND MINERAL PARTICLES FOR COAL PROCESSING
Researchers: Auro Lessa and Dr. Thomas Wheelock
Department: Chemical Engineering

Title: IMPROVED SORBENT UTILIZATION USING STAGED COMBUSTION
Researchers: Jeffrey Dykstra and Dr. Robert Brown
Department: Mechanical Engineering

Title: CALORIMETRIC CHARACTERIZATION OF FLY ASH FOR MINING AND ENGINEERING APPLICATIONS
Researchers: Argha Saha and Dr. John Pitt
Department: Civil and Construction Engineering

Title: POTENTIAL OF MINERAL BENEFICIATION PROCESSES FOR POOR-QUALITY LIMESTONE AND DOLOMITES
Researcher: Taher Z. Merchant and Dr. John M. Pitt
Department: Civil and Construction Engineering

Title: PREDICTING HANDLING AND DEWATERING CHARACTERISTICS OF DENSE-PHASE SLURRIES
Researchers: Steven Levorson and Dr. Robert Lohnes
Department: Civil and Construction Engineering

Title: A LAND USE AND LANDSCAPE PLAN FOR A NORTHERN MISSOURI COAL MINE
Researchers: Heidi Williams and Prof. Norman Dietrich
Department: Landscape Architecture

Miscellaneous Projects (1989-1990)

During the 1990 fiscal year, ISMMRRI provided seed money to support several short-term projects. Descriptions of each project are included in the following pages.

Title: INFLUENCE OF COAL AND SORBENT CHARACTERISTICS ON FLUIDIZED BED COMBUSTORS
Researchers: Dr. Robert Brown and Balmurli Natrajan
Department: Mechanical Engineering

Title: THE EFFECTS OF ADSORBED ORGANIC MATTER ON GYPSUM DEHYDRATION
Researchers: Dr. Robert Cody and Thad Slaughter
Department: Geological and Atmospheric Sciences

Title: EMPIRICAL MODELING OF AIR-BORNE DUST PARTICLES IN MINING OPERATIONS
Researchers: Dr. Audrey Levine and Mohamed Abuel-Kassem Mohamed
Department: Civil and Construction Engineering

In addition, the following researchers have industrial grants administered by ISMMRRI: Buttermore, Dawson, Dietrich, Lohnes, and Straszheim.

Title: SONIC ENHANCEMENT OF FINE COAL CLEANING BY CONVENTIONAL FROTH FLOTATION
Researchers: William H. Buttermore
Department: ISMMRRI

Title: A LAND USE AND LANDSCAPE PLAN FOR A NORTHERN MISSOURI COAL MINE
Researchers: Prof. Norman Dietrich and Heidi Williams
Department: Landscape Architecture

Title: COAL HANDLING CHARACTERISTICS
Researchers: Dr. Robert Lohnes and Steven Levorson
Department: Civil and Construction Engineering

Title: CHARACTERIZATION OF MINERALS AND SULFUR FORMS IN LIGNITE
Researchers: Dr. Warren E. Straszheim, Mark A. Nuetzmann, and Glenn M. Oren
Department: ISMMRRI and Civil and Construction Engineering

Student Research Projects

A REGIONAL FLUID INCLUSION STUDY OF THE CAVE-IN-ROCK FLUORITE DISTRICT, ILLINOIS[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

Greg Fuhrmann. Graduate Student
Department of Geological and Atmospheric Sciences

and

Dr. Paul G. Spry
Department of Geological and Atmospheric Sciences

As Part of the Allotment Program
of the Iowa State Mining and Mineral Resources Research Institute
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[1] This material is based upon work supported by the Bureau of Mines, U. S. Department of the Interior, under Grant #G1194119.

A REGIONAL FLUID INCLUSION STUDY OF THE CAVE-IN-ROCK DISTRICT, ILLINOIS

Greg Fuhrmann and Dr. Paul G. Spry
Department of Geological and Atmospheric Sciences

Background

The Cave-in-Rock District of southern Illinois and northern Kentucky is the largest fluorspar producer in the United States, with small amounts of zinc and lead. The area has many ore bodies of both vein and stratabound type. Fluid inclusion studies on some of the deposits have been completed by other researchers, but a regional study that ties in all of the mines of the district has not been done to date.

Objectives

The main goal of this research is to obtain fluid inclusion data from a large number of fluorite, calcite, quartz, sphalerite and barite deposits in the region and use the information to answer to some degree the following questions:

1. What are the relative differences in age, size, mineralogy, and conditions of formation between vein and stratabound deposits?
2. The structure of the district is complex with a dome, narrow grabens, and crosscutting faults. How did the various structural elements affect the deposition?
3. Typical Mississippi Valley-type deposits have extensive dolomitization, with very little fluorspar. Why is the Cave-in-Rock district so different in composition, and what is the origin of the fluorspar?
4. Most of the fluorspar is banded. Does the sequence of banding change within ore bodies? Does it change between ore bodies? How much does the homogenization temperature of the individual bands change in the region?

Progress

Fluid inclusion data is available from recent work on several stratabound ore bodies including the Deardorff, Hill, Minerva, and Denton mines (Richardson and Pinckney, 1984; Koellner, 1987) as well as on the Dyer Hill mine, a vein deposit (Richardson, 1985). The Denton mine lies northwest of the other stratabound ore bodies and is separated by a major fault-bounded graben.

During the fall semester, the Annabel Lee deposit which lies in the same area as the Denton deposit, was studied. Data collected along with observations by Clark (1988) on the Annabel Lee deposit indicate homogenization temperatures of early fluorites (Y2 band) from 111° to 139°C; salinities range from 20.1 to 22.6 equivalent weight percent NaCl. The homogenization temperatures of Denton mine Y2 fluorites range from 117° to 134°C, with salinities 20.8 to 22.2 percent NaCl (Richardson and Pinckney, 1984).

The early purple (P3) bands in the fluorite crystals were lower overall and narrower in variation in homogenization temperatures compared to the Y2 fluorites of each mine, with the Annabel Lee mine temperatures in the 121.5° to 127°C range and from 120.6° to 140.8°C for the Denton mine (Koellner, 1987). The Dyer Hill homogenization temperatures for P3 fluorite ranged from 116.0° to 127.7°C (Richardson, 1985). The salinities of the Annabel Lea and Denton mines were 21.4 to 21.8 and 20 to 23 equivalent weight percent NaCl, respectively (Clark, 1988; Koellner, 1987).

During the spring semester, research concentrated on analyzing fluid inclusions in samples from vein deposits. The mines investigated included Alcoa, Crystal Mt., Henson, Rock Candy Mt., and Victory. Rock Candy Mt. samples are of very dark purple fluorite making it impossible to clearly see any inclusions. The samples from the other mines are clear to yellow fluorite, with minor purple banding.

The paragenetic sequence visible in fluorite from stratabound deposits is difficult to positively correlate to vein deposits. Many of the samples from vein mines are of one color and/or lack characteristic purple or blue banding. Also, the banding seen in vein samples may not be related to the banding in stratabound fluorites. Since the paragenetic sequence of the vein deposits has not been determined with a satisfactory level of confidence, the naming of zones for inclusions measured may be subject to change.

Primary fluid inclusions from the Victory mine had higher temperatures than the other deposits, in a narrow range from 146.0° - 148.1°C. The inclusions are in what appears to be the P3 zone of the paragenetic sequence. Fluid inclusion temperatures from the Crystal Mt. mine ranged from 134.7° to 144.8°C. Alcoa mine fluid inclusion temperatures were much lower and had a greater range, from 91.0° to 127.9°C. No primary inclusions were found from the Henson mine samples studied.

The secondary fluid inclusion temperatures generally had a greater range than the primary temperatures. For example, Crystal Mt. secondary inclusions ranged from 113.3° to 127.0°C, and measurements from Alcoa were from 100.3° to 121.6°C. Henson mine temperatures for secondary inclusions were higher and in a smaller range of 138.0° to 143.9°C.

Salinities for all fluid inclusions, primary or secondary, in all mines studied were similar, and ranged from 19.04 to 20.74 equivalent weight percent NaCl for primary inclusions and from 19.46 to 21.75 equivalent weight percent NaCl for secondary inclusions.

The data collected so far does not indicate a significant difference in fluid composition between vein and stratabound deposits. The temperatures are lower in mines further away from Hick's Dome, but the salinities group close together.

Future Work

The fluid inclusion measurements will be completed during the summer and fall of 1990. The information obtained will be used to determine relationships between stratabound and vein deposits, if any. One main goal in the coming year will be to find any vertical and/or horizontal temperature gradients within the mines in the fluorspar district.

Plans are to finish writing a computer software program that will be able to read data files of fluid inclusion data and to obtain characteristics of the fluid, such as NaCl content, molality, and mole fraction, as well as to calculate the internal pressure at the time of homogenization as well as density, specific volume, and molar volume.

Presentations

Fuhrmann, G. D. and P. G. Spry, "A preliminary fluid inclusion study of Illinois-Kentucky fluorspar district," Iowa Academy of Science, April 20-21, 1990, Drake University, Des Moines, Iowa, V. 97, p. A-34.

References

Clark, D., "Fluid inclusion data for Annabel Lee Fluorite and Sphalerite," unpublished report, 1988.

Koellner, M.S., "A study of the fluid inclusion, stable isotope and mineralogical characteristics of the Denton fluorspar deposit, Cave-in-Rock, Illinois," unpublished thesis, Iowa State University, 1987.

Richardson, C.K., and Pinckey, D.M., "The chemical and thermal evolution of the fluids in the Cave-in-Rock fluorspar district, Illinois: mineralogy, paragenesis, and fluid inclusions," Econ. Geol., Vol. 79, p. 1833-1856, 1984.

Richardson, C. K., unpublished data, 1985.

GEOCHEMICAL MODELING OF THE ARCHEAN LODE GOLD DEPOSIT AT JARDINE-CREVASSE MOUNTAIN, MONTANA[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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and

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As Part of the Allotment Program
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GEOCHEMICAL MODELING OF THE ARCHEAN LODE GOLD DEPOSIT AT JARDINE-CREVASSE MOUNTAIN, MONTANA

Ping Liu and Dr. Paul G. Spry
Department of Geological and Atmospheric Sciences

Background and Objectives

Gold deposits spatially associated with Precambrian iron formation are of particular importance worldwide. Among the most important examples are the Homestake deposit, South Dakota, the Kolar deposit, India, and the Lupin deposit, Canada. Like other deposits associated with sedimentary rocks, the origin of gold deposits associated with iron formation is controversial. Three main genetic models have been proposed for these gold deposits: a syngenetic model involving simultaneous deposition of gold and the iron formation; an epigenetic model involving a later introduction of gold, arsenic, sulfur and probably calcium and silicon into the iron formation; and a multistage model requiring a primary concentration of gold during the formation of iron formation and a remobilization and reconcentration of gold during later events. The Archean iron formation-hosted Jardine lode gold deposit, Montana, was selected to evaluate these models.

Although over 55% of the world's gold production has been directly or indirectly derived from rocks of Archean (>2.2 Ga) age, relatively few Archean lode gold deposits have been found in the United States. They are the Jardine deposit (Montana), the Atlantic City-South Pass deposits (Wyoming), and the Ropes deposit (Michigan). The fact that the Archean, Jardine and Atlantic City-South Pass deposits, as well as the Proferozoic, Homestake deposit (South Dakota), are situated in the Wyoming Province suggests that the Province may be a Precambrian gold province similar to Precambrian (Archean) Provinces in Canada (e.g., Abitibi Province). The development of an exploration model from the study on the Jardine gold deposit is very useful for the search of further similar gold deposits in the Wyoming Province.

Work Accomplished

Microscopic studies. Reflected light studies show that there are four types of arsenopyrite in the gold ores from the Jardine deposit: skeleton crystals, crystals with solid inclusions, small euhedral crystals, and large crystal aggregations. Microprobe analyses reveal that their chemical compositions, represented by the As/S ratio are different. Two types of amphiboles are found in the rocks from the Jardine deposit by transmitted light studies. One is hornblende, and another is cummingtonite. Microprobe analyses confirmed the observation.

Microprobe analyses. Microprobe analyses have been done on the four types of arsenopyrite and the two types of amphiboles. Table 1 shows that the As/S ratios of the rims are higher than those of the central portions, and the ratios for small grains are larger than those for large aggregations. From Table 2, we can see that the amphibole in the rocks containing sulfides is hornblende, with a low Mg+Fe/Ca+Al ratio, whereas the amphibole in the rocks free of sulfides is cummingtonite, with a very high Mg+Fe/Ca+Al ratio.

Table 1. Compositions of Arsenopyrite from Jardine Deposit, Montana.

Occurances		Pts	As Content (wt%)		S Content (wt%)		As/S Ratios	
			Range	Ave.	Range	Ave.	Range	Ave.
Skeleton Crystals	Rims	28	47.6-51.0	49.2	16.7-18.8	18.0	2.56-3.02	2.73
	Tiny Grains	3	49.5-50.3	49.9	16.7-17.8	17.4	2.80-3.01	2.87
Crystals with Solid Inclusions	Rims	6	49.0-52.0	50.6	17.4-18.8	18.1	2.63-2.94	2.79
	Central	5	45.3-48.5	46.5	18.6-20.5	19.6	2.22-2.61	2.37
Small Euhedral Crystals		26	45.8-49.4	48.4	16.9-19.1	18.1	2.55-2.85	2.67
Large Crystal Aggregations		9	45.0-48.6	46.7	17.5-19.0	18.2	2.44-2.78	2.57

Table 2. Compositions of Amphiboles from Jardine Deposit, Montana.

Occurances	pts	Element Content (wt%)							Mg+Fe/Ca+Al
		Si	Al	Fe	Mg	Ca	Na	K	
Interstitial among large arsenopyrite grains	9	40.64	17.91	22.43	4.42	11.05	1.29	0.49	0.93
Associated with arsenopyrite skeletons	4	41.13	18.85	21.93	4.22	11.21	1.28	0.44	0.87
No sulfides in the assemblage	4	51.22	0.90	38.27	7.00	0.68	0.03	0.06	28.7

Fluid inclusion studies. Fluid inclusion studies of quartz in iron formation at Jardine show the presence of two basic types of inclusions at room temperature (approximately 25°C):

Type 1 - CO_2-bearing inclusions, which contain discernible CO_2 indicated by the appearance of a CO_2-dominant fluid phase and a gaseous mixture of CO_2 and H_2O in the inclusions.

Type 2 - Aqueous inclusions, which do not contain discernible CO_2 in the inclusions but may contain one or more daughter crystals.

Heating and freezing experiments yielded similar results to the fluid inclusions in quartz from other Archean lode gold deposits (Table 3).

Future Work

Future work includes further field, fluid inclusion, and isotope studies and electron microprobe analyses.

Deposits	CO_2-Bearing Inclusions				Aqueous Inclusions			References
	Tm-co2	Tm-clath	Th-co2	Th-total	Tm-hydrate	Tm-halite	Th-total	
Jardine, Montana	-65- -58	5 - 6	25- 28	210-240	-25- -13	> 190	140-180	This study
Atlantic City-South Pass, Wyoming	-55- -60	-12- 22	-12-30	170-430	-51- - 8		50-310	Spry & McGowan 1989
McIntyre-Hollinger, Timmin, Canada	-58- -54	9- 12		200-350	-18- 2		160-390	Smith et al., 1984
Pamour, Porcupine, Canada	-66- -59	-15- 0	11- 16	195-380	-20- -2		90-190	Walsh et al., 1988
Sigma, Abitibi, Canada	-63- -56	4- 11	-32-31	180-395	-45- -25	140-235	60-285	Robert & Kelly, 1987
Wawa, Canada	-58- -55	7- 15	15- 30	220-360	-18- -7		120-220	Studemeister & Kilias, 1987

Table 3. Characteristics of Fluid Inclusions from Various Archean Lode Gold Deposits.

Publications

Liu, P. and Spry, P. G., "The geological setting of the Archean Jardine lode gold deposit, Montana," J. Iowa Acad. Sci., V. 97, p. A33, 1990.

Spry, P. G., McGowan, K. I., and Liu, P., "A fluid inclusion and stable isotope study of Archean lode gold mineralization in the Wyoming Province, U.S.A.: International Conference on Mineral Deposit Modeling," 8th Symposium of the International Association of the Genesis of Ore Deposits, in press, 1990.

References

Robert, F. and Kelly, W. C., "Ore-forming fluids in Archean gold-bearing quartz veins at the Sigma Mine, Abitibi greenstone belt, Quebec, Canada," Econ. Geol., 82, p. 1464-1482, 1987.

Smith, T. J., Cloke, P. L., and Kesler, S. E., "Geochemistry of fluid inclusions from the McIntire-Hollinger gold deposits, Timmins, Ontario, Canada," Econ. Geol., 79, p. 1265-1285, 1984.

Spry, P. G. and K. I. McGowan, "The origin of Archean lode gold deposits, Atlantic City-South Pass district, Wyoming, U.S.A.: a fluid inclusion and stable isotope study," 28th International Geological Convention Abstracts, 3, p. 163-164, 1989.

Studemeister, P. A. and Kilias, S., "Alteration pattern and fluid inclusions of gold-bearing quartz veins in Archean trondhjemite near Wawa, Ontario, Canada," Econ. Geol., 82, p. 429-439, 1987.

Walsh, J. F., Kesler, S. E., Duff, D., and Cloke, P. L., "Fluid inclusion geochemistry of high-grade, vein-hosted gold ore at the Pamour mine, Porcupine camp, Ontario," Econ. Geol., 83, p. 1347-1367, 1988.

GEOCHEMICAL STUDY ON THE GIES GOLD-TELLURIDE DEPOSIT, JUDITH MOUNTAINS, MONTANA[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

Xiaomao Zhang, Graduate Student
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As Part of the Allotment Program
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GEOCHEMICAL STUDY ON THE GIES GOLD-TELLURIDE DEPOSIT, JUDITH MOUNTAINS, MONTANA

Ping Liu and Dr. Paul G. Spry
Department of Geological and Atmospheric Sciences

Background

This research is a geochemical study of the Gies gold-telluride deposit, Montana. The Gies deposit is located within an alkalic intrusive complex (monzonite, quartz monzonite and syenite) with the gold-telluride mineralization occuring in quartz veins.

Objectives

The proposed research will investigate:

a. The formation temperature and pressure of the gold-telluride ore and the quartz veins;

b. The nature and the origin of the fluids, which are responsible for the deposition of the ores;

c. The physical and chemical environment of ore formation;

d. Mineralogy of the ore system;

e. Genetic relationship between ores and igneous intrusive rocks; and

f. From the above data, a genetic model for ore formation.

To achieve the above objectives, the following geological and geochemical studies will be performed:

a. Field work to collect necessary geological data and samples;

b. Microscopic (reflected and transmitted light) studies of the mineralogy of ore and vein minerals and the petrology of host rocks;

c. Electron microprobe studies of hard-to-identify minerals, especially gold-silver tellurides;

d. Fluid inclusion studies, including heating and freezing experiments on vein-forming minerals, mainly quartz;

e. Stable isotopic studies, including oxygen, hydrogen, carbon and sulfur isotopes, on vein and ore minerals and host rocks;

f. Trace element studies of the igneous host rocks; and

g. Thermodynamic studies of mineral assemblages and aqueous tellurium species at elevated temperatures.

Progress

During the last year, the following progress has been made.

Field Work. The field work at the Gies mine last summer was mainly conducted on the ore and quartz vein system; vein and host rock samples were collected.

Fluid Inclusions. There seems to be at least two generations of primary fluid activity (shown by different generations of quartz veins). These fluids consist of NaCl and H_2O and have very similar properties (salinities, etc.). The two recognized stages of fluids include:

(a) Homogenization temperature of 280°-300°C, with a salinity of 5 to 8 wt. % NaCl equivalent;

(b) Homogenization temperature of 210°-230°, with a salinity of 5 to 8 wt. % NaCl equivalent.

A fluid with similar salinities might also occur at temperatures of 240°-250°C; however, considerable more data are required to verify this conclusion.

Secondary inclusions show homogenization temperatures at 170°-180° and 130°-140°, respectively.

Mineral Identification. The following minerals were identified in vein materials:

Silicates: quartz, roscoelite, muscovite, kaolinite;
Carbonates: calcite, dolomite;
Oxide: hematite
Sulfides: pyrite, galena, sphalerite, marcasite, chalcopyrite, bornite;
Sulfosalt: tetrahedrite;
Tellurides: sylvanite, krennerite, hessite;
Others: fluorite.

Microprobe Analyses of Minerals.

(1) Gold-Silver Minerals

Three phases have been detected: Sylvanite, krennerite and hessite. Sylvanite is probably the most abundant gold-silver mineral in the Gies Mine. Some typical analyses are listed in Table 1.

Table 1. Wt. % of Sylvanite, Krennerite, and Hessite in Gies Mine Samples.

Sample	Mineral	Au	Ag	Te
G-6	Sylvanite	30.02	8.39	61.01
M-1-1	Sylvanite	26.39	11.07	62.38
P-16-2	Sylvanite	23.54	12.66	62.98
G-6	Krennerite	36.61	4.11	59.25
G-5	Hessite		62.59	37.88

(2) Roscoelite

Roscoelite analyses show that the compositions vary considerably. Generally, this mineral is mainly composed of Si, Al, V and K. The K content is relatively uniform (total range of 7.54 - 9.73%) in different samples whereas the Si, Al and V contents change considerably. Some typical analyses are listed below in Table 2:

Table 2. Roscoelite Composition - Wt. % Oxides.

Sample	Si	Al_2O_3	Cr_2O_3	V_2O_5	FeO	MgO	CaO	K_2O	Total
M-1	44.61	11.06	0.72	26.88	0.20	1.81	0.21	8.74	94.21
G-1	45.47	7.51	0.96	29.91	0.06	1.39	0.20	7.88	93.39
P-5	39.04	19.30	0.66	23.19	0.37	1.47	0.17	8.73	93.08

Stable Isotope Analyses

Seventeen quartz samples, representing different types and stages of veins, were separated for oxygen isotope analyses. The results show that their $\delta^{18}O$ values are relatively uniform, with most values in the range of +15 to +17 per mil (the total range is from +13.4 to +18.1 per mil). With the knowledge of the formation temperatures (from fluid inclusion data) of these samples, the calculated $\delta^{18}O$ values of the fluids that were responsible for the vein precipitation are +5 to +7 per mil. These values indicate a strong magma component to the ore-forming fluid.

Sixteen sulfide samples (galena, sphalerite and pyrite), separated from vein materials, showed fairly uniform $\delta^{34}S$ values, ranging from -1.0 to +3.1 per mil, which are consistent with a magmatic origin.

Future Work

In the coming year, research will concentrate on the following aspects:

a. A field trip to Gies Mine during June 1990 to collect representative samples of all igneous phases existing in mine area, including monzonite, quartz monzonite and syenite;
b. Detailed microscope studies on: (1) relationships among ore and gangue minerals; (2) mineral paragenesis; and (3) alterations;
c. Further electron microprobe studies of gold-silver tellurides and some other unidentified minerals;
d. Fluid inclusion studies of different quartz veins;
e. Oxygen isotope studies of host rocks, mainly igneous intrusive rocks;
f. Hydrogen isotope studies on selected hydrothermal minerals (kaolinite and roscoelite);
g. Trace element analyses of intrusive rock, by using x-ray fluorescence spectroscopy; and
h. Thermodynamic studies on mineral assemblages.

Presentations

Xiaomao Zhang, "Oxygen Isotope Geochemistry of Witwatersrand Pelitic Rocks, South Africa," J. Iowa Academy of Science, V. 97, p. A-33, 1990.

LEACHING OF SPHALERITE-CONTAINING ORES[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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Any opinions, findings, conclusions, or recommendations expressed in this publication are those of the authors and do not necessarily reflect the views of the Bureau of Mines, Department of the Interior.

LEACHING OF SPHALERITE-CONTAINING ORES

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Objective

The purpose of this project is to develop a practical leaching method for extracting zinc from sphalerite-containing ores. Preferably, this method should avoid roasting or pyrometallurgical processing, and the attendant environmental pollution problems, and result in a solution that is ready for electrochemical processing.

Progress

A sphalerite-containing ore from New Mexico was subjected to a number of leaching tests with acidic peroxide solutions. A petrographic analysis of the ore revealed the following composition: 35-62% sphalerite, 15% chalcopyrite, 6-10% pyrite, 2-4% marcasite, and 10-40% gangue minerals. The ore was ground to pass a 200-mesh screen, and an analysis of the ground material by energy-dispersive x-ray fluorescence indicated the following distribution of elements: 31.5% Zn, 19.0% Fe, 5.4% Cu, 1.5% Mn, 23.3% S, and 6.15% Si.

For each leaching test, 10 g of ground ore was treated in a stirred flask with an aqueous solution of the leachant at a given temperature for 1 hour. The extract was analyzed for zinc by atomic absorption spectrophotometry and the undissolved solids were dried and weighed.

The peroxides employed for most of the leaching tests were potassium monopersulfate and t-butyl hydroperoxide. The potassium monopersulfate was obtained from the DuPont Company as the triple salt $2KHSO_5 \cdot KHSO_4 \cdot K_2SO_4$ produced under the name of Oxone. The organic hydroperoxide was obtained as a 70% aqueous solution from Aldrich Chemical Company.

The results of a number of leaching tests are presented in Table 1. The following parameters were varied among these tests: temperature, type of peroxide, peroxide concentration, and sulfuric acid concentration. The percentage of zinc extracted from the ore is reported. The best results were achieved in run K22 when over 90% of the zinc was extracted at room temperature with a solution containing 0.35 $\underline{M}$ Oxone and 1.0 $\underline{M}$ sulfuric acid. Generally, better results were achieved with Oxone than with t-butyl hydroperoxide. It required much higher concentrations of t-butyl hydroperoxide than of Oxone to achieve similar levels of extraction. The percent extraction increased with peroxide concentration up to a point. In some ranges of Oxone concentration, zinc extraction was improved by the presence of sulfuric acid. However, there appeared to be a complex interaction between peroxide concentration and sulfuric acid concentration. With a 2.0 $\underline{M}$ solution of t-butyl hydroperoxide, the zinc extraction actually decreased when the sulfuric acid concentration was raised above 0.5 M. The results seemed to be affected only slightly by temperature.

Table 1. Results of leaching sphalerite ore with acidic peroxide solutions.

Run No.	Temp., °C	Peroxide Type	Peroxide M	H_2SO_4 M	Tot. Vol., ml	Zn Extracted, %
K1	20	Oxone	0.10	0	250	26.8
K12	20	Oxone	0.20	0	250	51.6
K16	20	Oxone	0.30	0	250	65.6
K20	20	Oxone	0.35	0	250	79.4
K30	20	Oxone	0.35	0	250	80.4
K31	20	Oxone	0.45	0	250	88.3
K28	20	Oxone	0.10	1.0	250	41.7
K14	20	Oxone	0.20	1.0	250	65.6
K18	20	Oxone	0.30	1.0	250	85.4
K22	20	Oxone	0.35	1.0	250	90.3
K29	20	Oxone	0.45	1.0	250	85.3
K2	90	Oxone	0.10	0	250	27.8
K13	90	Oxone	0.20	0	250	51.6
K17	90	Oxone	0.30	0	250	76.4
K21	90	Oxone	0.35	0	250	72.5
K32	90	Oxone	0.45	0	250	78.4
K25	90	Oxone	0.10	1.0	250	45.6
K15	90	Oxone	0.20	1.0	250	78.4
K19	90	Oxone	0.30	1.0	250	79.4
K26	90	Oxone	0.30	1.0	250	77.4
K23	90	Oxone	0.35	1.0	250	83.3
K27	90	Oxone	0.45	1.0	250	78.4
T11	40	t-butyl	0.54	0.46	135	24.5
T8	60	t-butyl	0.54	0.46	135	26.2
T2	80	t-butyl	0.54	0.46	135	29.2
TT1	60	t-butyl	1.50	0.50	250	57.1
TT2	60	t-butyl	2.00	0.50	250	74.1
TT3	60	t-butyl	2.50	0.50	250	74.4
TT4	60	t-butyl	3.00	0.50	250	74.4
TT5	60	t-butyl	2.00	1.00	250	63.5
TT6	60	t-butyl	2.00	3.00	250	58.7
TT7	60	t-butyl	2.00	5.00	250	49.2

Future Work

Due to the complex interaction between peroxide concentration and sulfuric acid concentration, more leaching tests will be conducted to determine the optimum combination of these concentrations. Also, consideration will be given to the extraction of copper and the disposition of other elements such as iron.

HEAT OF IMMERSION OF COAL AND THREE-PHASE CONTACT ANGLE IN COAL-OIL-WATER SYSTEM[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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[1] This material is based upon work supported by the Bureau of Mines, U. S. Department of the Interior, under Grant #G1194119.

HEAT OF IMMERSION OF COAL AND THREE-PHASE CONTACT ANGLE IN COAL-OIL-WATER SYSTEM

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Objective

The main objective of this work is to establish a proper method of characterizing the hydrophobicity of coal and associated minerals in order to predict the behavior of these materials in the oil agglomeration process for cleaning coal.

Generally, surface hydrophobicity is expressed in terms of the three-phase contact angle. A large contact angle indicates strong hydrophobic behavior, while a small contact angle represents weak hydrophobic behavior. When a contact angle is zero, the liquid spreads completely on the solid. The heat of wetting in water is also an indicator of hydrophobicity. A large heat of wetting indicates low hydrophobicity while a small heat of wetting indicates large hydrophobicity. Both indicators of hydrophobicity are being determined to see how well they correlate with each other and to see how well the agglomeration response of coal correlates with one indicator or the other.

Research Progress

Three-Phase Contact Angle Measurement

Three-phase contact angle measurements are being made for the oil-water-coal system by using the suction potential method in which water and then oil (e.g., heptane) are drawn through a bed of coal particles while the pressure drop across the bed and the volume of effluent are measured. The design of the apparatus used by previous workers has been modified and improved. With the modified apparatus, the reproducibility of the measurements is fairly good. Contact angle measurements have been made for graphite from Sri Lanka, Upper Freeport coal from Pennsylvania, No. 2 Gas Seam coal from West Virginia, and Upper Freeport coal from the Helvetia mine. The observed relationship between the three-phase contact angle and the rank of coal is consistent with the known hydrophobicity of these coals. The effect of particle size on three-phase contact angle is being investigated as well.

Heat of Immersion of Coal

The heat of immersion is a very promising indicator of hydrophobicity since it is much more reproducible than other indicators. However, several factors such as the moisture content of the sample, particle size, and liquid type affect the heat of immersion. Therefore, the relationship between the heat of immersion of coal and these factors is very important for establishing standard conditions for comparing different coals.

Samples of Illinois No. 6 coal and No. 2 Gas Seam coal from West Virginia were prepared for immersion in various liquids in a microcalorimeter. Preparation included grinding, separation into narrow particle size fractions, and adjusting the moisture content. Separation involved screening with standard test sieves using a mechanical shaker followed by further screening with a Sonic Sifter. The particle size distribution of each of the smaller size fractions was determined with a Microtrac particle size analyzer whereas the size distribution of each of the larger size fractions was determined by automatic image analysis. The moisture content of the various size fractions was adjusted either by vacuum drying at room temperature or by placing the samples in desiccators containing saturated solutions of different salts and allowing enough time for the samples to reach a constant weight. The moisture content of each coal sample was determined subsequently by drying a small portion of the sample in an oven at 110-120°C for 1.5 hr. The ash and total sulfur contents of each sample were also determined by standard methods.

The heat of immersion of the different coal samples in water, methanol, heptane, and hexadecane at 25°C was determined by a colleague, Y.-C. Hu, using a Tronac model 450 microcalorimeter. The heat of immersion was observed to depend on the type of coal and the type of liquid as well as particle size and moisture content.

By assuming that the heat of immersion per unit of total surface area wetted by the liquid is a constant for a given type of coal and a given liquid, the following relation was developed:

$$\Delta H = h \cdot S_e + x \cdot S_i \cdot h$$

where

ΔH = total measured heat of immersion, J/g.

h = heat of immersion per unit of actual wetted surface, mJ/cm^2.

S_e = external surface area, cm^2/g.

S_i = internal pore surface area, cm^2/g.

x = fraction of internal surface wetted.

For a given coal of a given moisture content immersed in a given liquid, x and h are constants. The h value can be used as an indicator for comparing the hydrophobicity of different coals without the bias of particle size. The x values for these two coals were estimated by using the S_i value determined by mercury porosimetry analysis. The S_i is 46.85 m^2/g for Illinois No. 6 coal and 29.13 m^2/g for No. 2 Gas Seam coal. The small x value is consistent with the special pore structure of these coals. The validity of the above linear model was confirmed by plotting ΔH vs. S_e for both types of coal which have been vacuum-dried. Furthermore, the linearity of the experimental data has also been confirmed by statistical analysis. The χ^2-test showed that the linear model can be accepted with 95% confidence. Application of linear regression to the data produced the values of $x \cdot S_i \cdot h$ and h and correlation coefficient R as presented in Table 1.

Table 1. Results of Regression Analysis.

Coal Type	Liquid Type	$x \cdot s_i \cdot h$, J/g	h, mJ/cm^2	x	R
Ill. No.6	Water	21.59	11.86	0.0389	0.972
	Methanol	33.20	19.07	0.0372	0.939
	Heptane	16.24	30.62	0.0113	0.926
	Hexadecane	10.21	12.53	0.0174	0.930
	Neohexane	12.10	12.51	0.0206	0.989
No.2 Gas Seam	Water	1.250	4.520	0.0095	0.974
	Methanol	4.619	9.243	0.0172	0.916
	Heptane	6.854	4.075	0.0577	0.956
	Hexadecane	2.749	6.634	0.0142	0.975
	Neohexane	2.643	5.304	0.0171	0.959

Presentations

1. Y.-C. Hu and Xiaoping Qiu, "Heat of Immersion of Coal", Third International Conference on Processing and Utilization of High-Sulfur Coals, Ames, Iowa, Nov. 14-16, 1989.

2. Y.-C. Hu, Xiaoping Qiu, and T. D. Wheelock, "Heat of Immersion of Coal in Various Liquids," Iowa Academy of Science 102nd Annual Meeting, Des Moines, Iowa, April 20-21, 1990, V. 97, p. A-29.

COAL BENEFICIATION UTILIZING TRIBOELECTRIC CHARGING IN A FAST FLUIDIZED BED[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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[1] This material is based upon work supported by the Bureau of Mines, U. S. Department of the Interior, under Grant #G1194119.

COAL BENEFICIATION UTILIZING TRIBOELECTRIC CHARGING IN A FAST FLUIDIZED BED

David Tucholski and Dr. Gerald M. Colver
Department of Mechanical Engineering

Objective

The objective of this research is to preclean finely ground coal by removing the pyrite containing sulfur using electrostatic separation above a fluidized bed. The particles are first triboelectrically charged in a fast fluidized bed before being separated by an electrostatic precipitator. Physical separation is made possible since the carbonacous matter charges positively and the pyrite charges negatively when contacting selected materials such as copper.

Progress

The progress made between July 1, 1989, through June 30, 1990, can be separated into four principle efforts. The first deals with a literature search, the second with the design and construction of a Faraday cage, the third with the design and construction of a circulating fluidized bed, and the fourth with the measurement of the charge developed on the particles in the circulating bed. These efforts will be reviewed below along with the future plans for installing the electrostatic precipitator.

Literature Research. The goal of the literature search was to investigate previus studies where fluidized beds have been used to triboelectrically charge particles for mineral separation as well as to obtain design information on circulating fluidized beds.

Some of the more important studies to this research have been conducted by Inculet et al. (1977, 1980, 1982, 1984, 1988), Anderson et al. (1979), and Bergougnow et al. (1977) in which a fluidized bed was used to triboelectrically charge particles prior to a separation step. The beds used were of the non-circulating type, although Inculet (1977) used a dilute phase circulating loop to separate pyrite from coal. The loop was used only after the particles were charged in a 3-D fluidized bed. Kantz and Sears (1969) discussed electric field phenomena within fixed and fluidized beds.

Several books on the subject of circulating beds have been reviewed. These books give the basic principles and design criteria for the construction of a circulating bed, for example, Geldart (1986), Basu (1985, 1988), and Howard (1989).

Faraday Cage. To obtain data for the charging process that occurs in the fluidized bed, a specially designed Faraday cage was constructed. The Faraday cage is a rugged yet extremely sensitive and convenient instrument for measuring the net charge on the particles. Since the Faraday cage measures only the total charge on the captured particles, the charge/mass ratio is determined by dividing the charge by the mass of the sample. The Faraday cage allows for an evaluation of the effectiveness of the charging mechanisms in the fluidized bed, for example, by using different charging materials. Also, the design of the electrostatic precipitator (length, separation distance, electric field strength) for separation of the pyrite is determined in part by a knowledge of the charge/mass ratio of the particles (see Figure 1).

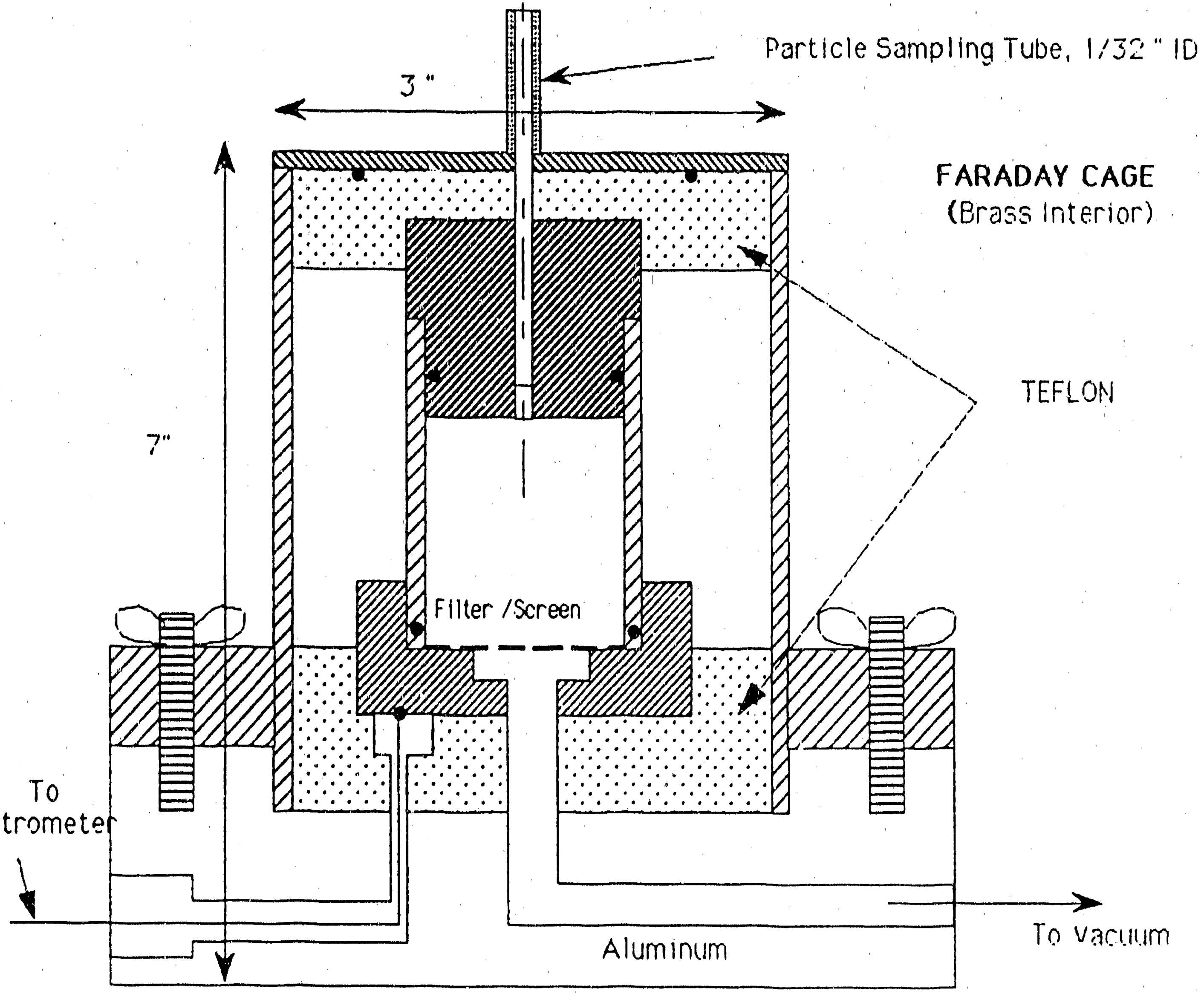

Figure 1. Faraday Cage for Measurement of Particle Charge/Mass Ratio.

It was necessary to design the Faraday cage so that it could be conveniently emptied and cleaned while maintaining an air-tight seal within the environment when in use. In addition, the Faraday cage itself as well as the sampling probe must be shielded from extraneous electric interference (such as body motion, etc.) by placing the cage and the probe within a second cage which is grounded.

The sampling probe proved to be difficult to fabricate as insulation was required to separate the 1/32 inch i.d. probe from its 3/32 inch i.d. shield. This meant that the probe had to be shaped into a convenient "U" bend (see Figure 2) after the tubes were put together and insulated with either Teflon or silicon glue. However, bending of the tubes generated a battery effect from stressing and rubbing of the insulation which generated currents of the same order of magnitude as the current being measured from the particles (i.e. $\sim 10^{-8}$ A). This problem was finally overcome by perfecting the probe assembly process.

Once perfected, a special calibration test was devised to determine if the Faraday cage was measuring charge correctly. A spherical particle 4.38 mm in diameter was affixed to a glass hypodermic needle (to minimize the contact area) and charged in a parallel plate capacitor of known electric field strength by touching one of the plates. The charged particle was then touched to the inner probe of the Faraday cage, and the indicated charge was compared against the theoretical Maxwell charge Q_t from the capacitor given by the equation:

$$Q_t = 4\pi\epsilon r^2 E(1.64)$$

where ϵ is the permittivity of free space, r is the radius of the particle, and E is the electric field strength. Because of the extreme sensitivity of the Faraday cage, it was found that correct readings could be made only if the probe itself was a large distance from the capacitor. Table 1 summarizes average readings (both positive and negative) taken over several scales on the electrometer.

Table 1. Charge on 4.38-mm copper particle.

Q_t (theory)	Q_a (measured)
5.25×10^{-10}	5.54×10^{-10} (Coulombs)

From these results, we concluded that the Faraday cage was working properly. However, a disadvantage of the Faraday cage is that it can only measure a net charge on the captured particles. This means that a separate test will be needed to sort out plus- and minus-charged particles.

<u>Circulating (Fast) Fluidized Bed</u>. Figure 2 shows the preliminary design of the circulating (fast) fluidized bed now undergoing testing in the Particulates and Combustion Laboratory. This initial bed was fabricated from Plexiglass to allow for visual observations of the flow characteristics of the bed. It is expected that a second bed will be constructed largely from copper because of its superior charging characteristics with pyrite (Anderson et al., 1979). To date, three different distributors have been designed to test the charging characteristics of the particles. These include swirling flow as well as straight flow throughput from below the bed.

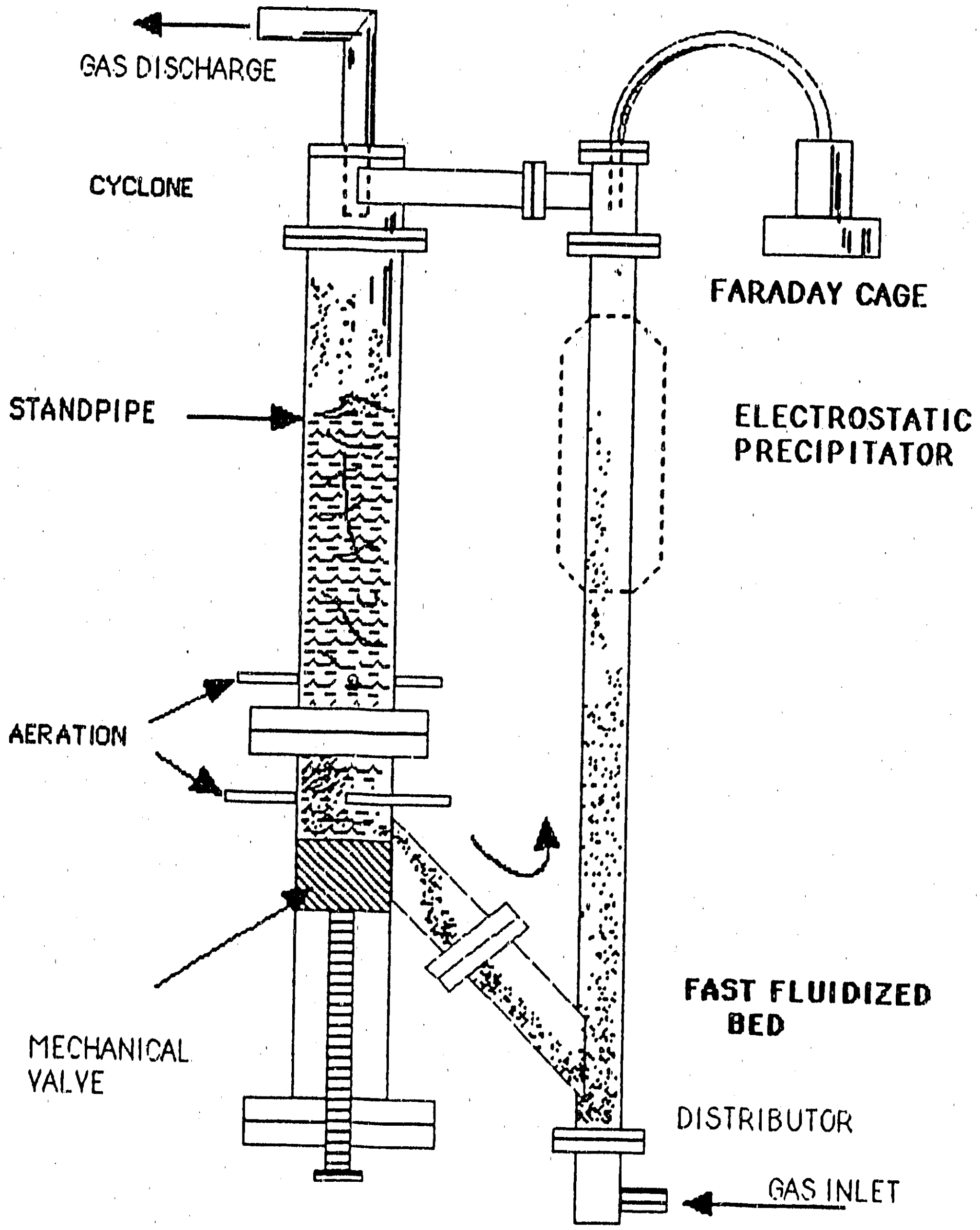

Figure 2. Circulating (fast) fluidized bed with electrostatic precipitator.

Preliminary runs have been made mainly with glass beads of 150 μm diameter which have worked reasonably well with the present design. However, "flowability" of the particles is of primary concern in such a system, and it is well known that finer particles exhibit cohesive effects that interfere with the hydrodynamics of the system. It is our goal to work with coal particles as small as 30 μm.

A feed problem was also encountered while operating the bed in the circulating mode. Initially, an L-valve was used as a non-mechanical valve to control the particle concentration above the fast fluidized bed. It has been shown (Basu 1988) that non-mechanical valves work best with granular Geldart Group B materials. Since we will be working with Geldart group A or C materials, the L-valve was changed to an angular feed system with a mechanical valve (Figure 2). This resulted in improved feed control. To reduce the problem of slug flow in the standpipe region of the bed (the left side in Figure 2), a larger diameter pipe than on the right side was used. This allowed for the aeration and fluidization in the standpipe for smooth flow through the valve. However, it is anticipated that additional flow control may be required for the smaller particles.

It is noteworthy that while the circulating bed can be run smoothly for long periods of time (e.g., 15-20 minutes), the effect of high charging in the bed is accumulative on the walls and on the particles themselves. The result is that electrostatic effects can be an important factor in controlling particle feed through the circulating bed loop (electrostatic buildup is common in flowing particulate systems often resulting in visual and audible displays of discharge). To help offset these effects, additional aeration tangentially above the valve was introduced to help break up the electrostatic forces between the particle and the wall. Humidification can also be introduced to control the electrical conductivity of the particles (and the walls) which in turn affects triboelectric charging (which is controlled by the Fermi level difference between contacting materials, surface and volume conductivity, grain or crystal structure, speed of contact, etc.).

<u>Particle Charge Measurement/Initial Coal Testing</u>. Glass particles were tested in the bed for charge measurement. The purpose of the test was to substantiate that the Faraday cage could be used to sample particles essentially of the same electrostatic sign. Tests showed that when sampling particles of high concentrations (visually), the electrometer measured high charges of typically 10^{-7} C/g compared to charge measurement at low particle concentrations of about 10^{-9} C/g. However, it was discovered that particle accumulation on the tip of the sampling probe was contributing to the higher charge/mass ratio readings taken at high particle concentrations. This test demonstrates that great care must be taken in charge sampling, especially at the higher particle concentrations.

Preliminary testing using powdered coal was also tried in the bed without any special design adjustments being made for the reduced particle size of about 50 μm (i.e., because of the cohesion of coal powder). These tests were basically inconclusive as the walls of the circulating bed became blackened making visual observations of performance impossible. It was felt that the apparatus operated successfully for at least a short period of time.

Future Plans

The next and last major device to be designed and tested in this research is the electrostatic precipitator which is used to capture and separate the pyrite from the coal. The precipitator must be sufficiently large to provide the residence time necessary for capture of the charged particles on opposite plates and also be designed for continuous cleaning in the steady-state operation of the bed. The designing and building of the precipitator will be undertaken during the summer of 1990. The precipitator will be placed initially in the fast fluidized bed section of the circulating bed. Following insertion of the precipitator, it is expected that a copper interior in the bed will be added to aid particle charging and also that different distributors will be tested as noted above.

Summary

To date, a great deal of effort has been put into this project in designing, testing, and building a successful Faraday cage and a circulating (fast) fluidized bed. Virtually all of the fabrication and testing of this project has been carried out by Mr. Tucholski (in addition to his research into the subject). It is observed that operating a circulating bed in a steady state mode requires a balance between bed hydrodynamic forces and (short range) van der Waals type, electronic cohesive forces especially with fine particles, and (long range) electrostatic forces resulting from triboelectric charging between the particles in the bed and the walls. The goal of this investigation is to achieve high particle charge using fine particles. It is anticipated that utilization of the electrostatic precipitator may help smooth the operation of the bed in that the most highly charged and smallest particles will be removed from the system. This means that some of the problems discussed above may be alleviated or at least reduced.

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CRYSTAL/GRAIN ALIGNMENT DURING PROCESSING OF YTTRIUM-BARIUM-COPPER PEROVSKITE (1-2-3) SUPERCONDUCTORS[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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As Part of the Allotment Program
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[1] This material is based upon work supported by the Bureau of Mines, U. S. Department of the Interior, under Grant #G1194119.

CRYSTAL/GRAIN ALIGNMENT DURING PROCESSING OF YTTRIUM - BARIUM - COPPER PEROVSKITE (1-2-3) SUPERCONDUCTORS

Timothy M. Green and Dr. Mufit Akinc
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Background

Common ceramic-forming processes such as extrusion and tape-casting tend to cause alignment of morphologically anisotropic particles. This alignment is such that the drag of the particle is minimized, which generally results in presentation of a surface of minimal area to flow of the plastic ceramic precursor. Using a process developed in our laboratory, a paste of controllable viscosity may be produced from a yttrium solution and salts of barium and copper. This paste, when calcined, yields the $YBa_2Cu_3O_{6+x}$ (1-2-3) superconducting phase. Because conduction in the 1-2-3 material is maximized in the a - b plane of the crystal, utilization of the material as a conductor will necessitate development of grain-aligned bodies. We will attempt to obtain grain alignment by seeding the precursor paste with anisotropic 1-2-3 powder, followed by extrusion of the paste-powder mixture.

Progress

Major impetus this period has been directed toward development of optimized precursor paste for the extrusion process. Initial extrusion experiments showed that it is possible to extrude fibers of a precursor made of yttrium nitrate solution, molten barium hydroxide, and crystalline copper nitrate. The precursor has a 1:2:3 stoichiometry with respect to the Y, Ba, and Cu ions. However, it was noted that the granularity of the paste limits ability to extrude long, continuous fibers. The granularity also contributes to stoppage of extrusion prior to full stroke. As such, chemical determination of the phases present in the green fiber was undertaken.

X-ray analysis of the ternary system shows that the green fiber consists of barium nitrate ($Ba(NO_3)_2$), copper nitrate hydroxide ($Cu_2(OH)_3NO_3$), and an x-ray amorphous yttrium phase. Scanning electron microscopy (SEM) and energy-dispersive x-ray analysis (EDS) indicates that the barium nitrate phase exists mainly as large (0.1 mm) crystals. Because the extruded fiber is only 1.0 mm in diameter, it is assumed that the large barium nitrate crystals are the cause of the tendency for the green fibers to fracture, and contribute to bridging which may stop flow of the extrudate during extrusion processing.

In order to see if it would be possible to extrude fibers of a 1-2-3 precursor system which did not contain barium nitrate, an acetate-based system was tried. Stoichiometric amounts of barium hydroxide, yttrium nitrate solution, and copper acetate were mixed and excess water evaporated to obtain a workable viscosity. The extruded fiber obtained is uniform in cross-section, and the full load of paste could be extruded without stoppage. X-ray analysis of the green material shows that the phases are again barium nitrate, copper nitrate hydroxide, and an amorphous yttrium phase. Firing of the green material yields the 1-2-3 superconducting composition as the principle phase.

Future Work

Due to the presence of barium nitrate as a product in the "acetate-based" system, it has been decided that a yttrium acetate solution or solid will be used in a series of future experiments rather than the previously used yttrium nitrate solution. The effect of this change on the rheology of the system will be examined. The rheological behavior of a paste containing a chloride-based yttrium compound will also be determined. Once a uniform, workable paste is obtained, the effect of adding 1-2-3 oxide powder to the paste will be examined in terms of grain alignment of the particles and in terms of effect on viscosity. Additional work in the next period will include an attempt to develop a model to describe the degree and mechanism of alignment of the grains.

In the past semester, Tim Green has accepted a position as a graduate research assistant for the Ames Laboratory. He is working on the completion of his thesis.

ENHANCEMENT OF LOW-QUALITY LIMESTONE AND DOLOMITIC AGGREGATES FOR USE IN CONSTRUCTION[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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ENHANCEMENT OF LOW-QUALITY LIMESTONE AND DOLOMITIC AGGREGATES FOR USE IN CONSTRUCTION

John Vu and Dr. John Pitt
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Objective

A majority of mining income is derived from industrial minerals used primarily in civil engineering construction. Typical applications include concrete aggregate, where the resulting composite must be strong and resistant to environmental deterioration including frost action and chemical reactivity. Nevertheless, some stones are found to be extremely resistant while others are short-lived because of the composition and the character of the voids and chemical contaminants. The objective of the project is to find ways to enhance the value of low-quality limestone and dolomitic aggregates. Inexpensive treatment involving fly ash will be attempted.

Background

In order to evaluate the potential for the interaction among aggregate type and deicer compounds, four categories of coarse aggregate and six brine compositions were selected. The four aggregates and their anticipated roles in the experiment are as follows:

1. **Alden**: This is a Class 2 or 3 aggregate, depending on bed. It has a good field service record and a corresponding response to accelerated freeze-thaw testing. The Alden aggregate is nearly pure calcium carbonate with a porous structure.

2. **Garrison**: This is a Class 2 aggregate with an intermediate field service record. It has shown good response to accelerated laboratory testing. Its dolomitic composition with traces of pyrite and its open pore structure make this stone suspect for chemical interaction with deicers.

3. **Waucoma**: This is a Class 3 aggregate of nearly pure calcium carbonate with a closed pore structure and a good service record. This aggregate was selected because its composition is similar to the Alden stone but with a different pore structure.

4. **Ames Mine**: This is a Class 2 aggregate of nearly pure calcium carbonate that performed moderately well in accelerated freeze-thaw tests. This aggregate has a pore structure of intermediate size (between Waucoma and Alden). Its chemical composition is similar to that of the Alden and Waucoma aggregates.

Mercury intrusion porosimetry tests were conducted to verify the pore structure characteristics of the aggregate samples used. The results of these tests are summarized in Table 1. While the cumulative pore size distributions were found to hold with the general character of each stone, a better quantitative comparison of pore sizes can be made from the median pore sizes listed at the bottom of the table. To summarize: media size, or one-half of the pores in the Alden and Ames Mine aggregates, were either larger or smaller than 5,500 and 3,000 angstroms, respectively. The median for the Garrison sample exceeded 10,000 angstroms. In contrast, the Waucoma stone's median was only 120 angstroms.

Table 1. Pore Size Distribution of Aggregates.

Pore Radius (Angstroms)	Alden	Garrison	Waucoma	Ames Mine
	(Cumulative Percentage of Pore Volume)			
10,000	16	57	0	1
5,000	53	72	8	5
2,000	68	83	12	71
1,000	72	88	13	83
500	75	90	18	89
200	80	93	31	92
100	84	96	60	95
50	90	98	80	97
20	98	99	98	99
	(Median Pore Size, Angstroms)			
	5,500	10,000	120	3,000

Composition of freeze-thaw solutions used in tests are identified and listed in Table 2. Solutions 1 and 6 are controls. Solution 2, with the impurity of magnesium chloride, was formulated because of preliminary results from tests on mortar where it was once thought to be a worst-case condition. This was later found not to be the case. Solution 3 was fabricated as representing a worst-case sulfate concentration. Solution 4 is a sample typical of Kansas salt sources. Solution 5 is the Kansas salt modified with calcium chloride to represent sand-salt de-icing mixtures commonly used.

Table 2. Brine Compositions Used in Concrete Freeze-Thaw Testing.

	(Weight Percent by Solute)					
Solution	NaCl	$MgCl_2$	$CaCl_2$	$CaSO_4$	$MgSO_4$	Comments
1	100.0	0	0	0	0	Control
2	98.0	2.0	0	0	0	Synthetic
3	98.0	0	0	2.0	0	Synthetic
4	96.1	0.2	0	3.61	0.09	Kansas Salt
5	94.6	0.2	1.57	3.55	0.09	Kansas Salt + $CaCl_2$ sand
6	0	0	0	0	0	Control

Methods

ASTM C-666 freeze-thaw specimens were cast from an Iowa DOT C-3 concrete mix, with Sioux City fly ash replacing 15% of the portland cement. The concrete mix had a water-cement ratio of 0.43, an 1.5-inch slump, and a 6% air content. The specimens were also cured for 28 days in accordance with ASTM C-192. The six brines were introduced in the freeze-thaw pans by maintaining the brine level at the beam's mid-height. Measurements of dynamic modulus of elasticity and pulse velocity were made at about every 30 freeze-thaw cycles. In addition, the flexural strength was determined after 300 cycles by 1/3 point loading.

Durability Factor

Table 3 summarizes the average durability factor for three repetitions of each test variable. Interpretation of these data are as follows:

Alden: Concrete made with this stone just met the lower expectation for Class 2 aggregate tested in water (Durability Factor, DF = 80%).

When fly ash was included in the Alden mix, a significant and consistent improvement was observed for all of the solutions. The fact that fly ash produced a significant improvement for all of the brines suggests that mortar is an important factor. The porous aggregate offering a conduit to the mortar could also contribute to deterioration.

Garrison: Tests in water with this sample of Garrison aggregate produced a lower durability factor than indicated by experience (DF = 67%).

An increased durability factor occurred with fly ash concrete in the natural brines, but slight reductions were observed with the synthetics.

While there may be some sensitivity to types of brines, adding fly ash was found to have a mixed influence on frost resistance. This suggests an aggregate-brine interaction rather than an attack on the mortar.

Waucoma: Concrete with this aggregate is consistent with previous experience when tested in water (DF = 89%).

Sensitivity to brine composition is not apparent and use of fly ash corresponds to a consistent increase in durability factor, also without regard to type of brine.

The importance of a non-porous aggregate pore structure on freezing and thawing in a deicing environment is indicated by these data. The difference between this and the two stones previously discussed is closed versus open pore structures. Open pores in the aggregate may be the conduit by which deleterious brines are allowed to make contact with the mortar.

Ames Mine: Concrete for test slabs using this aggregate was purchased from a local ready-mix producer. All slabs contained fly ash (i.e., concrete without fly ash was not tested). The concrete performed better in water than experience suggests. Limited sensitivity to brines was observed, with the most severe degradation occurring for solutions 4 and 5, those with natural salt. These results support the observation that a closed pore system in aggregate can be beneficial to resisting deterioration. A median pore size of 3,000 angstroms performed well.

Table 3. Durability Factors With and Without Fly Ash (FA).

			Brine Solution Used			
Aggregate	Sol. 1	Sol. 2	Sol. 3	Sol. 4	Sol. 5	Sol. 6
Alden w/o FA	61	52	52	51	53	80
Alden w/FA	84	99	98	95	98	93
Garrison w/o FA	58	81	79	51	52	67
Garrison w/FA	68	72	75	60	81	76
Waucoma w/o FA	90	86	94	91	96	89
Waucoma w/FA	102	98	101	99	99	103
Ames Mines w/FA	96	96	95	90	92	99

Tensile Strength

An alternative appraisal of freeze-thaw performance with de-icers is flexural strength after three hundred cycles. Figures 1 through 4 are plots of strength versus solution for the aggregates included in this study. The average of these results correspond to those for durability factor, in that fly ash increased most in strength when used with the Garrison aggregate; fly ash was observed to have a lesser impact with Alden stone and caused minimum response with that from Waucoma.

The most significant result of this test is that fly ash reduces variability in strength when used with the Garrison aggregate. Some Garrison aggregate samples mixed without fly ash were found to have no flexural strength, an extreme condition; however, the average was close to the average for fly ash concrete.

When concrete made with the Waucoma and Ames Mines aggregates were treated with the natural brines, flexural strength was found to be minimum. Flexural strength tests were found to be ineffective at differentiating the effects of brine treatments for the other aggregates and brines.

Conclusions

Based on the limited data available for freeze-thaw deterioration of concrete with different types of coarse aggregate, the tentative conclusions are as follows:

> In addition to the mortar phase, the pore structure of coarse aggregate has a significant influence on the ability of concrete to resist freeze-thaw deterioration in the presence of sulfate-tainted rock salt brines.
>
> Porous aggregates used in this study were the most vulnerable, in that the de-icer brines produced sizable reductions in durability factor. Although altering the mortar phase with fly ash was beneficial, the amount of improvement depended on the chemical composition of the rock. Improvement was adequate with a limestone, but only marginal with a dolomite.
>
> A non-porous stone was least affected by the brines. Also, fly ash improved durability of a good quality concrete.

Future Work

John Vu has finished all of his graduate requirements and accepted a position with the Iowa Department of Transportation.

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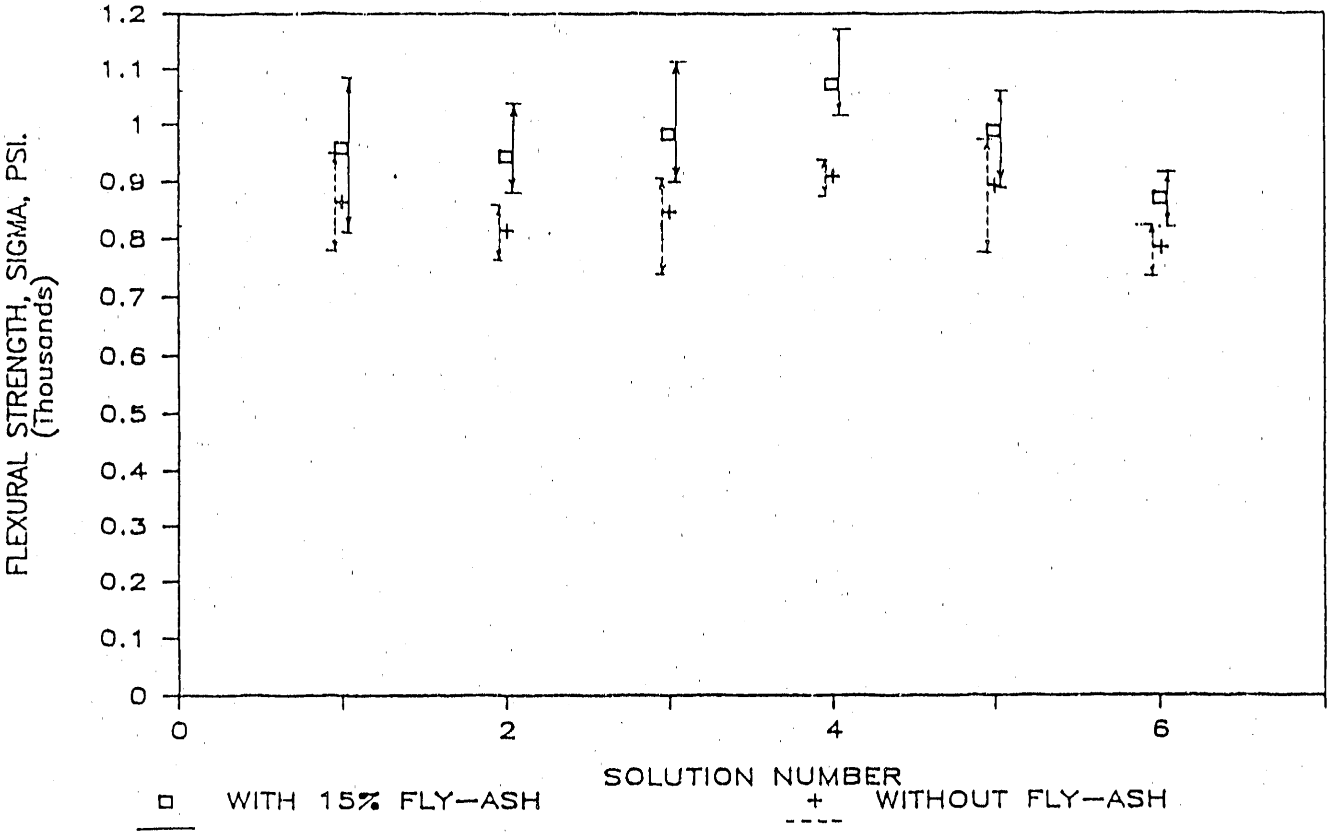

Figure 1. Flexural strength with Alden aggregate.

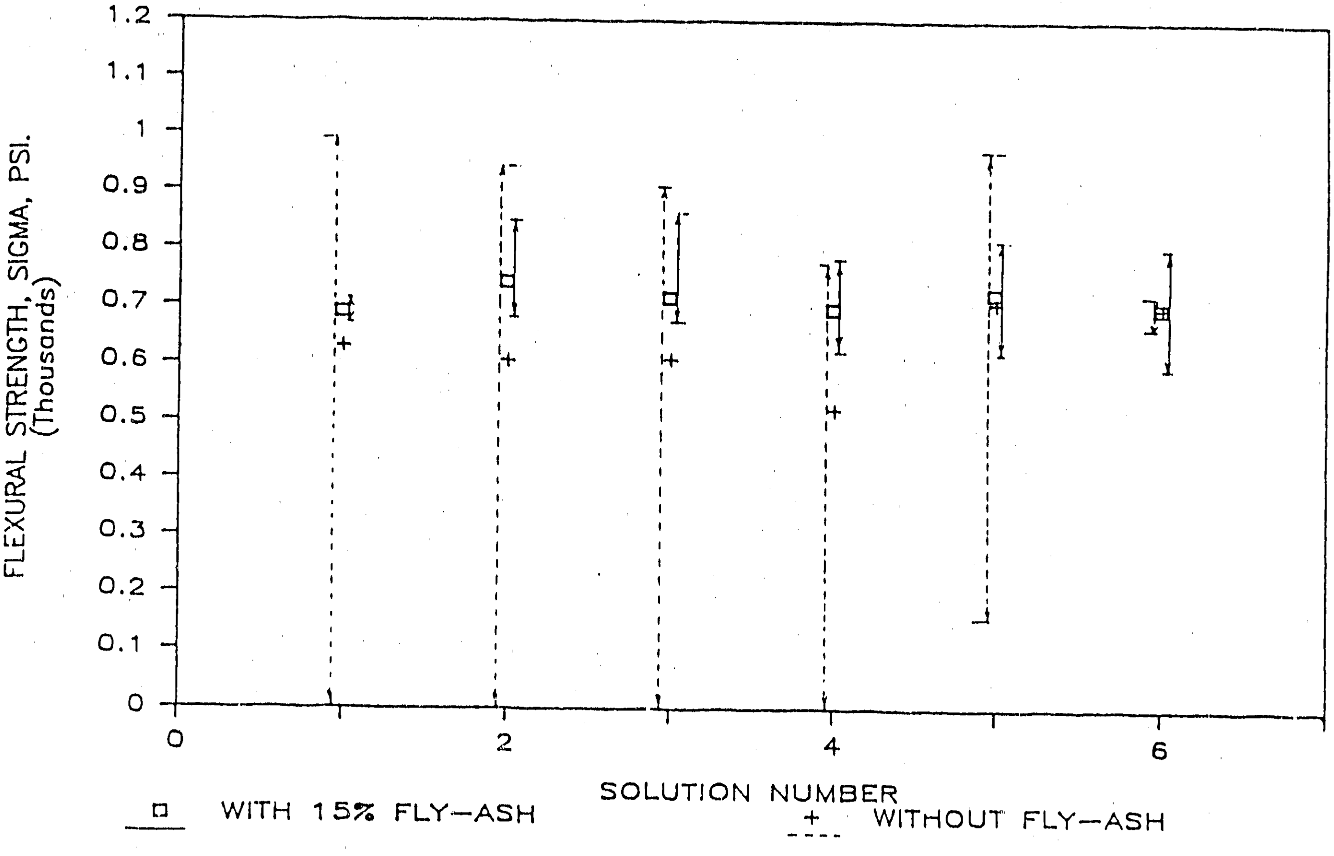

Figure 2. Flexural strength with Garrison aggregate.

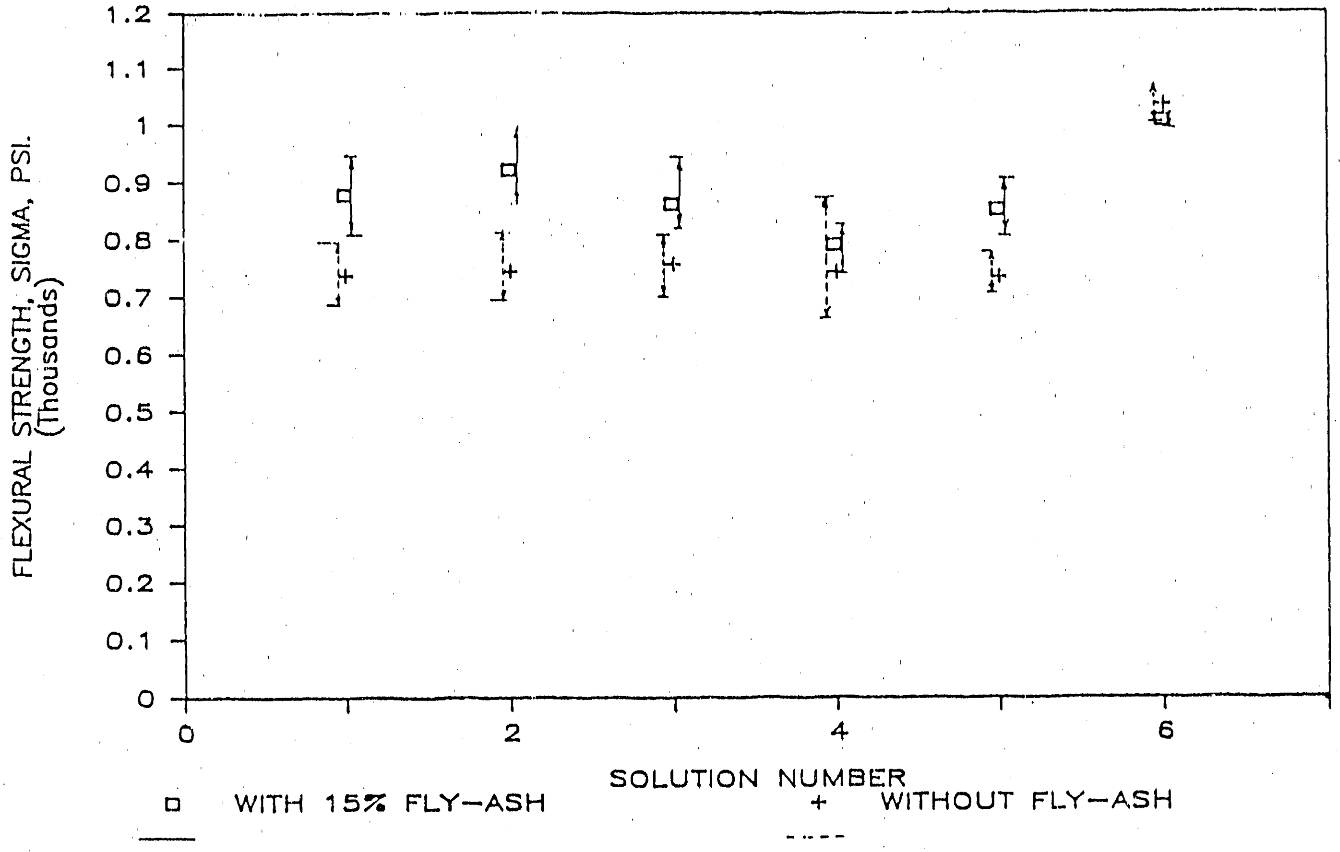

Figure 3. Flexural strength with Waucoma aggregate.

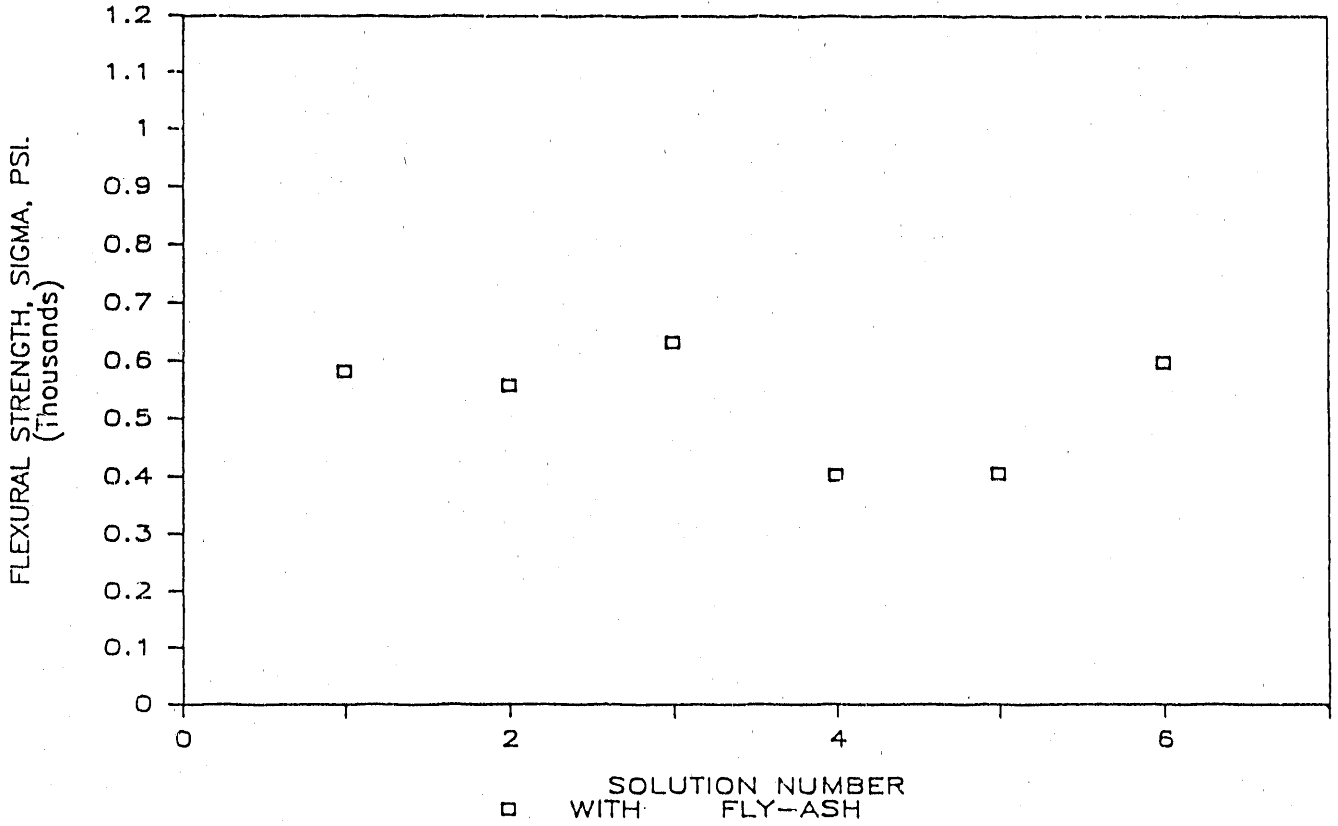

Figure 4. Flexural strength with Ames Mine aggregate.

THE IMPACT OF SURFACE MINING ON THE FT. DODGE, IOWA, AREA[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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THE IMPACT OF SURFACE MINING ON THE FT. DODGE, IOWA, AREA

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Objective and Background

This research stems from the lack of cooperation between planning officials and mining companies. The two are at opposite ends; instead of working together to achieve a common land use goal, they are working against each other. The mining companies are doing their best in reclamation efforts while staying within their financial limitations. The planning officials on the other hand, are doing their best to deter mining. They are continually passing more and more regulations that are not only restricting what the mining companies can and cannot do, but also placing extra financial burdens on the companies which in turn makes reclamation more difficult.

Mineral extraction offers great potential towards the development of landscape characteristics that will sustain creative reclamation land uses. It is these landscape and land-use potentials that planning officials are overlooking and subsequently eliminating through additional regulations rather than encouraging their development and capitalizing on them. There is a strong need for the development of common reclamation goals between planning and mining officials in order to benefit from these opportunities. The development and application of these common goals and a comprehensive plan is the main objective of this research. It is hoped that through accomplishing this objective several other connected objectives will also be made apparent to planning officials and the general public. These additional objectives are as follows:

- An awareness to the potentials of mineral extraction and reclamation activities as a force in the creation of beneficial and sustainable landscapes and land uses will be realized.
- The importance of planning and controlling mining and reclamation activities for the successful implementation and achievement of community land use goals will be illustrated.
- Community awareness of the opportunities and necessities of mineral extraction will be increased.
- The illustration of how mining may benefit a community will be made.
- The potential benefits of concurrent mining and reclamation planning to achieve the goals of both the quarry operators and the local community will be illustrated.

Progress

Throughout the spring semester, many hours have been spent gathering ecological and sociological base information of the Fort Dodge community and surrounding mining region. This base information consists of data concerning soil types, geological background, watershed information, watertable depths, drainage patterns, vegetation patterns, landform characteristics, topographic features, slope analysis, land ownership, land use, transportation, mineral resources and deposit locations, county, township, and section boundaries, future land use needs, mining permit information, mining methods being utilized, past-current-future mining locations and reclamation activities. It was determined that a graphic format utilizing a pin-bar and Mylar overlays would best suit this study. This format will allow for rapid and easy combination of different base maps to determine future land use and landscape characteristics limitations and opportunities of the area. Although the base information gathered this past semester remains the same, it will take a great amount of time to redraw the maps as Mylar overlays. Additionally, time has been spent researching literature sources to identify examples of reclamation projects and mining/land use regulations that have been implemented at other locations that may also apply to this research study.

Future Plans

This coming fall semester will be spent completing the Mylar base maps and attempting to gain information that identifies the views of both planning officials and mining operators concerning existing mining activities and regulation policies. This information is necessary to gain a better understanding of why a lack of cooperation between the two exists and what steps must be taken to create a positive working relationship.

Experience with Industry

The summer of 1989 was spent by Ms. Spoden as an intern in the Properties Division of the CalMat Company of Los Angeles, California. This internship allowed her the opportunity to gain experience in land planning and permitting and the development of post-mining land use plans.

The upcoming summer (1990) will be spent as an intern with the land planning firm of Florence-Martinez Associates of Tustin, California. Her time will be spent in the Natural Resources Division developing reclamation plans, Environmental Impact Statements, and collecting the information necessary for mining permit applications.

ON-LINE DETERMINATION OF UNBURNED CARBON IN AIRBORNE FLY ASH[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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ON-LINE DETERMINATION OF UNBURNED CARBON IN AIRBORNE FLY ASH

Jeffrey Dykstra and Dr. Robert Brown
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Objective

The objective of this research is to investigate the use of photoacoustic absorption spectroscopy (PAS) as a method for on-line monitoring of carbon in the exhaust gas of coal-fired boilers.

Background

Unburned carbon in fly ash is presently measured by a tedious and sometimes inaccurate procedure (ASTM Standard, 1982). Flue gas is sampled from the exhaust of a combustor, and a particulate filter is used to remove fly ash from the flow. The fly ash is dried and weighed before being placed in a furnace for several hours in order to burn off the carbon. The fly ash is then reweighed to find the percent carbon. This analytical procedure often consumes several hours. Rarely does filtering yield a large enough sample of fly ash to investigate transient phenomena in a combustor. The accuracy of this technique is limited by problems associated with isokinetic sampling and filtration techniques (Hawksley et al., 1977).

We are investigating photoacoustic spectroscopy (PAS) as a method for on-line monitoring of carbon in the exhaust gas of coal-fired boilers. Photoacoustic absorption spectroscopy is based on the periodic heating of a gas when amplitude-modulated radiation is absorbed by the gas or by particles suspended in the gas (Rosencwaig, 1980). This periodic heating produces an acoustical wave that can be detected by a microphone. Two important features of PAS make it especially suitable for detection of carbon in flue gas. The first of these is the ability of this technique to detect very weak absorptions. Secondly, the PAS signal is unaffected by light scattering in a particulate-laden gas flow; the acoustical signal arises only from light absorption. Consequently, only particles with complex refractive indexes, such as soot or char, contribute to the signal. Most mineral matter scatters light rather than absorbs it. Accordingly, PAS has good potential for distinguishing unburned carbon from mineral matter suspended in flue gas.

For low light absorption, the PAS signal (S) (Roessler and Faxvog, 1979) is given by:

$$S = RP_oA_AML$$

Where R is the cell responsitivity (dependent on cell geometry and light modulation frequency), P_o is the incident power, A_A is the integrated mass-specific absorption coefficient of suspended char particles, M is the mass concentration of absorbing particles, and L is the optical path length of the cell.

Experiments performed with synthetic fly ash confirm that PAS can distinguish unburned carbon from mineral matter in the fly ash (Brown and Dona, 1989). The present work will determine whether the technique can be applied to actual fly ash from an operating fluidized bed combustor.

Progress

Two PAS cells have been compared using NO_2 as a calibration gas. Figures 1a and 1b show a schematic of both cells. The redesigned cell should have a value of R eight times larger than that of the original cell. An improvement of only 30% was realized (see Figure 2 and 3). This could be due to the fact that the microphone area was significantly larger than the cross sectional area of the cell. Although the value of R was smaller than theoretically predicted, the signal-to-noise ratio was improved by a factor of over three.

On-line tests have been performed with the original cell using fly ash from a lab scale fluidized bed combustor. Noise from the violent nature of the combustion process traveled through the sample line and made it impossible to extract the photoacoustic signal from the total signal picked up by the microphone. With the redesigned cell and changes in the sample line configuration, this problem has been overcome. Using the new configuration, a photoacoustic signal of 32.0 microvolts was measured when the carbon mass loading was 0.45 g/m^3.

Future Plans

On-line tests will be run with the redesigned photoacoustic cell to determine whether the PAS signal is linear with carbon loading.

Jeff Dykstra will complete his M.S. thesis during the summer of 1990. He will then begin working towards his Ph.D. degree in Mechanical Engineering.

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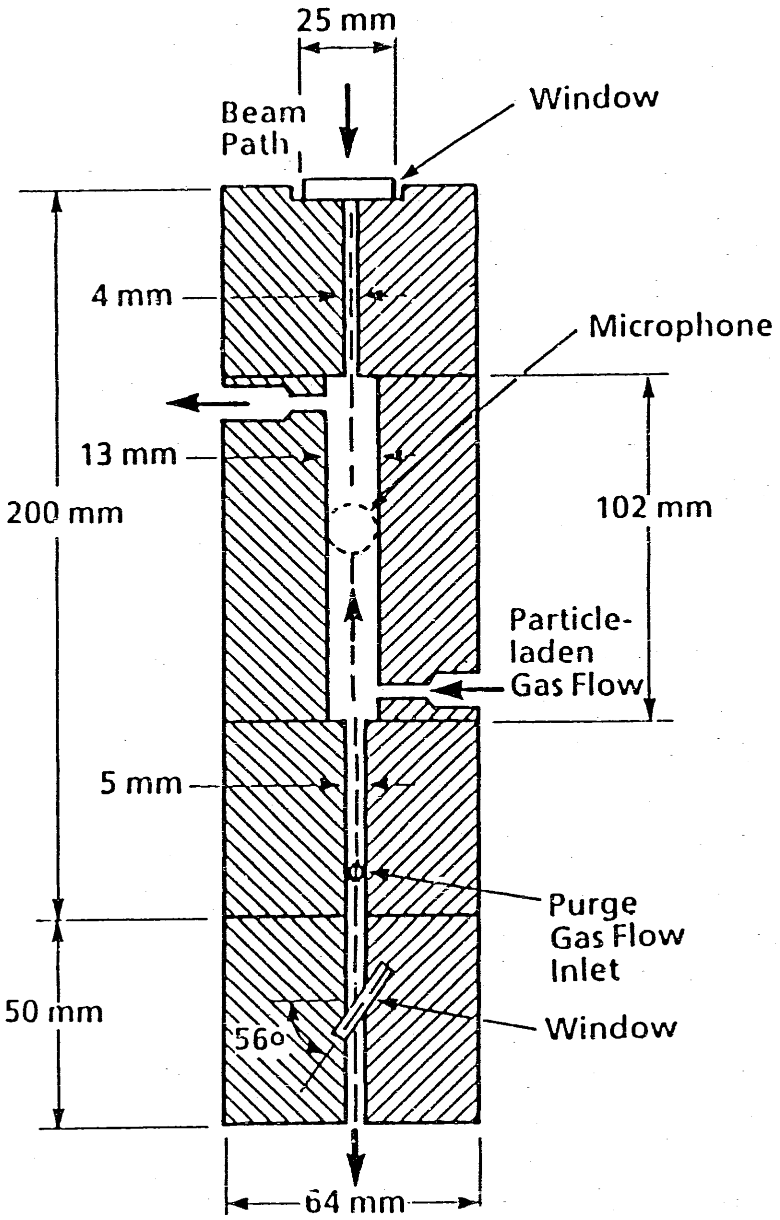

Figure 1a. Original Cell Design

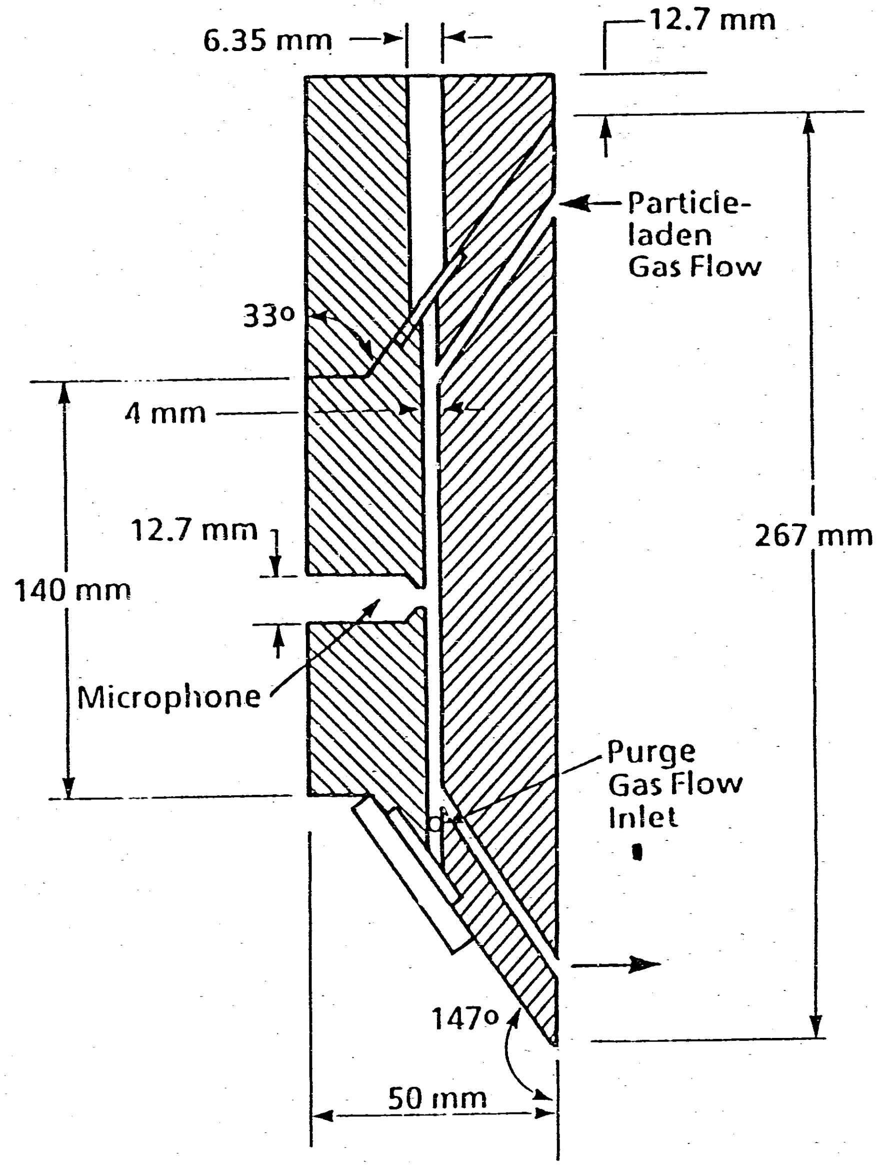

Figure 1b. Redesigned Cell Design

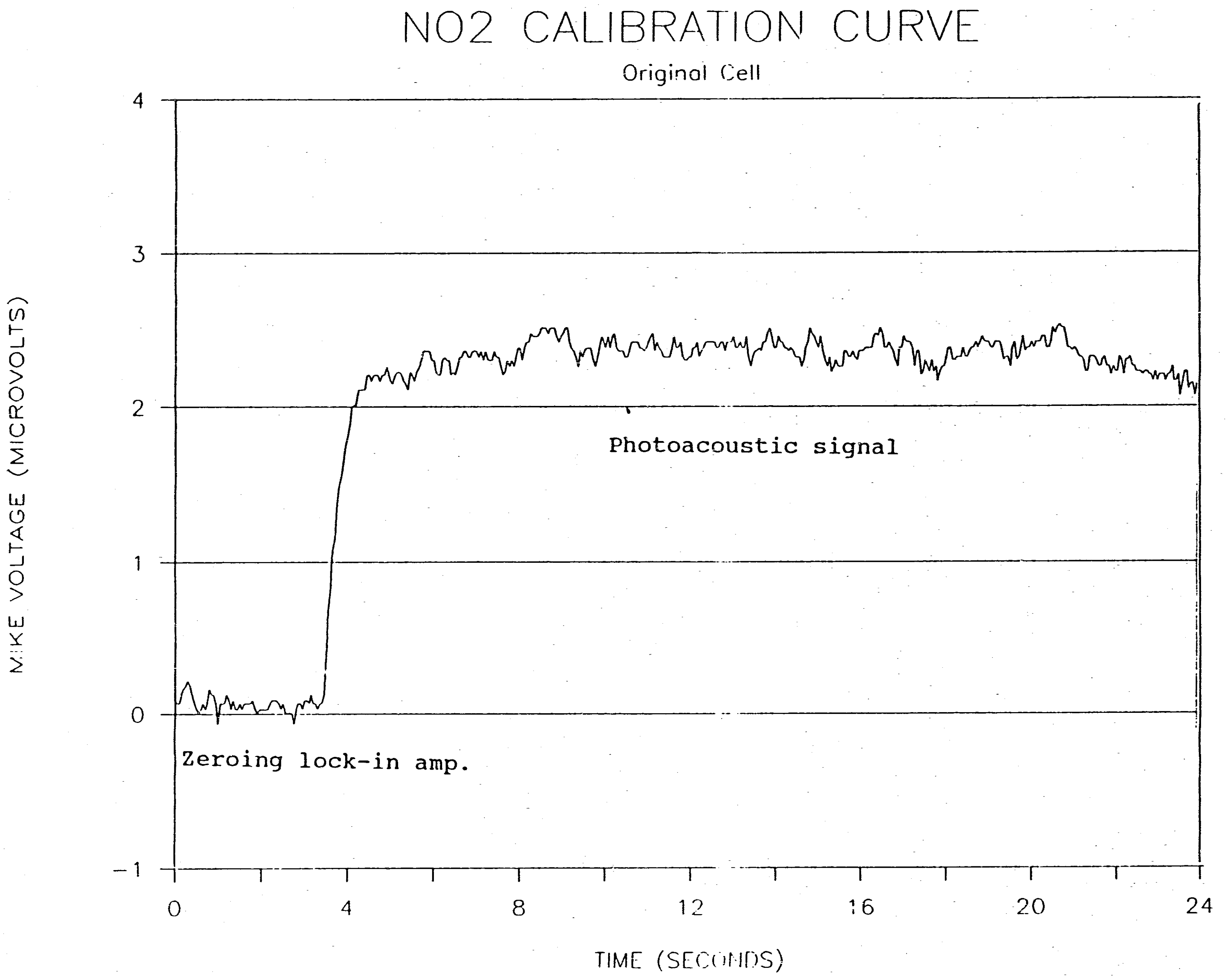

Figure 2. Calibration Signal of Original Cell

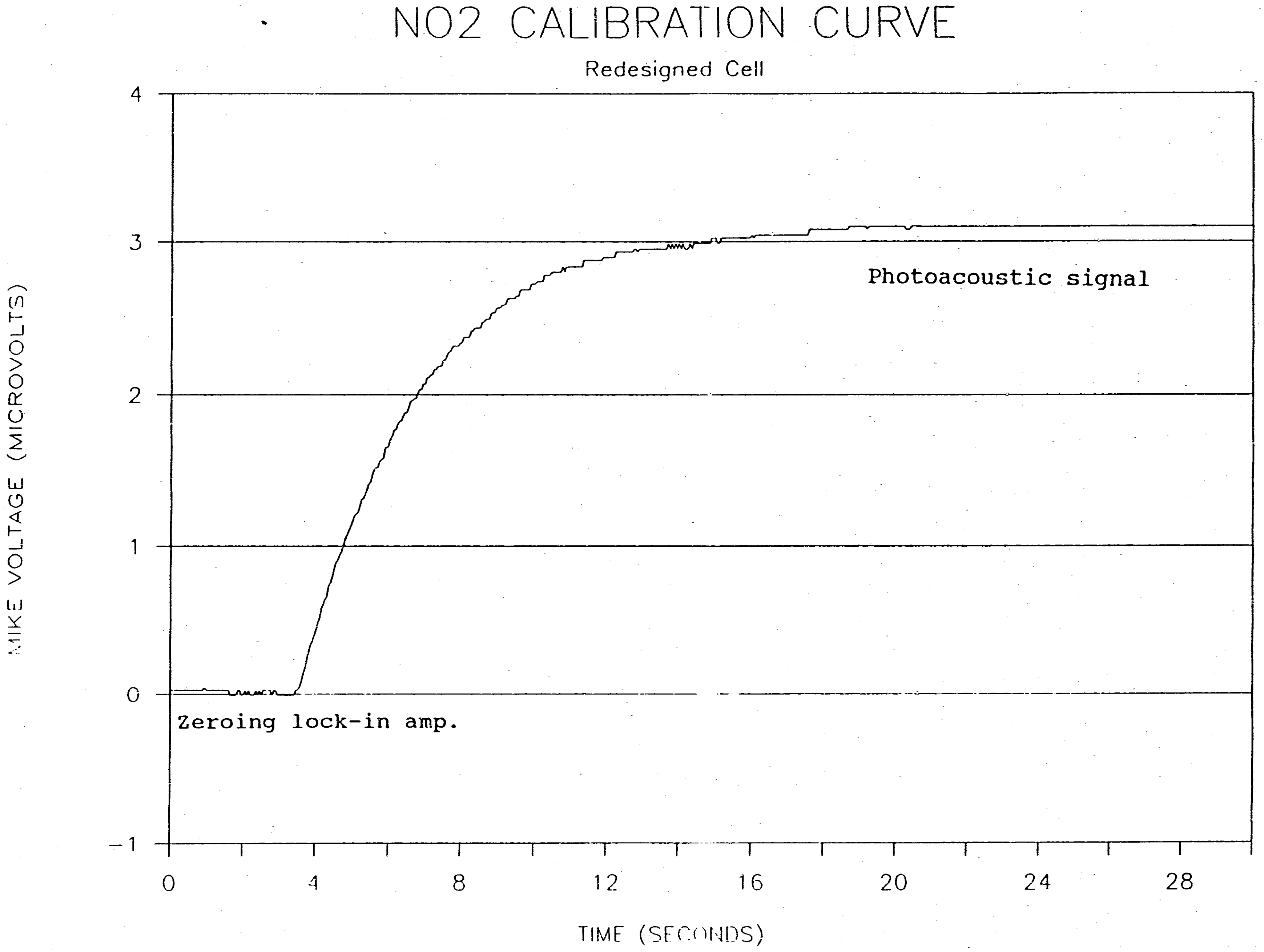

Figure 3. Calibration Signal of Redesigned Cell

CALORIMETRIC CHARACTERIZATION OF FLY ASH FOR MINING AND ENGINEERING APPLICATIONS[1]

Final Report for July 1, 1989 - June 30, 1990

Work Performed By

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CALORIMETRIC CHARACTERIZATION OF FLY ASH FOR MINING AND ENGINEERING APPLICATIONS

Argha Saha and Dr. John Pitt
Department of Civil and Construction Engineering

Objective

The purpose of this research is to explore an alternative method of fly ash characterization that is rapid, inexpensive, and rugged enough to be used under field conditions.

Introduction

Use of fly ash as a substitute for portland cement in concrete is a major application of the material; however, only 6% of U. S. production is currently being used (Idorn, 1982). Common practice is to limit fly ash substitution to around 15%. Intrinsic variability of fly ash, coupled with the complexity of its composition, gives a perception of uncertain outcome from higher fly ash substitution. However, in many cases, with substitution in the order of 50%, enhancements in both strength and durability properties have been observed. An effective, rapid, and simple characterization of fly ashes can assist the engineer in selecting the proper fly ash for a high fly ash-substituted concrete. A faster characterization technique is important because some components of fly ash hydrate very fast in the presence of little moisture, and if stored improperly they can change its properties drastically over a short period of time.

The pore solution of a cement fly ash paste is strongly alkaline, consisting of sodium and potassium hydroxides, which can dissolve the glassy phase of a fly ash. Also, the amount of silica and alumina eluted from a fly ash glass by alkalies has a direct relationship to the compressive strength of a fly ash cement paste (Uchikawa et al., 1986). These ideas can be utilized in the characterization of fly ashes. The dissolution of fly ash glass in a strongly alkaline medium and consequent formation of hydration products can be studied by solution calorimetry. These reactions have a direct relation to what takes place in a cement fly ash paste and will be helpful in predicting the performance of a fly ash in such medium. The solution calorimetry techniques help to identify the reactivity of a fly ash glass in terms of heat evolution in a strongly alkaline medium. Other than the glassy phase, the cementitious crystalline components may react with available water in the system and thus interfere with the heats involved in the reaction of glassy phase alone. An estimation of the self-cementitious fly ash components can also be done from the heat evolution of fly ashes in water. Extended laboratory analyses are required to check if the reaction products are hydrated calcium silicates or aluminates and whether the fly ash glassy spheres are taking part in the reaction.

Experimentation

Fly ashes from 7 different sources and silica fume were selected for evaluation. According to the ASTM classification, Neal #2 (N#2) and Neal #3 (N#3), are Class F and the remainder, i.e., Neal #4 (N#4), Ames (AM), Ottumwa (OTM), Clinton (CL) and Weston (WES), are Class C fly ashes. X-ray fluorescence (XRF) was used to determine elemental composition. Clinton and Weston fly ashes were analyzed by x-ray diffraction (XRD) in their unreacted, hydrated, and reacted states to monitor compound formation.

Hydrated and reacted Clinton fly ash was analyzed by thermogravimetric (TGA) and differential thermal analysis (DTA) to identify and compare the amounts of hydration products.

A 6-unit calorimeter was built wherein 6 different reactions could be run simultaneously and temperature fluctuations recorded. Each unit is made up of a Dewar flask and a Nalgene plastic container which is nonreactive to alkalies and is a good insulator. As shown in Figure A, the plastic containers are equipped with a mechanism to keep the fly ash from reacting with the solution inside the container by a controlling mechanism to trigger the reaction. These containers are set in Dewar flasks which are put in a styrofoam box to provide good insulation. The box has facilities to access the controlling mechanism in each of the calorimeter units and to allow thermistor wires to get into each unit. Any temperature fluctuation inside a calorimeter is recorded by a thermistor which is attached to a PC with an interphase and necessary software to record the data at a preset time interval. Temperature readings from each of the six calorimeters are simultaneously recorded in the PC. Temperature sensitivity of the setup is of the order of 0.01°C. Test runs were undertaken to make sure that the calorimeter units can operate under a reasonably adiabatic condition and that necessary insulation was provided.

Each of the fly ashes (20 g) was reacted in calorimeters with 200 g of water and 2N NaOH separately, and the temperature rise was recorded for up to 800 min. From the data available on crystalline composition, a simulated sample was prepared with maximum values of each component that would be present in a 20-g sample, except for the tricalcium aluminate (C_3A). The reason for not using C_3A was the unavailability of a pure compound. The simulated sample was reacted with 2N NaOH and the temperature rise was recorded for 800 min.

The heat capacities of the calorimeter units were measured by reacting 1 g of hydroxymethylaminomethane and 200 g of 0.1 N HCl and recording the temperature rise. The heat evolution in such a reaction is 117.5 cal at 25°C. Since the heat evolved was known, it was divided by the temperature rise to get the heat capacity. The measured temperature rises were found to vary by 5%. In any hydration and reaction with NaOH using a particular fly ash, the six calorimeters recorded identical patterns of temperature rise with a fluctuation of around 10%. In view of this, the variation of heat capacities among the calorimeters was neglected.

Fly ash samples were stored in airtight containers. For a reaction test, 20-g samples were measured into the plunger pan (Fig. A) of each of the calorimeter units. Reacting solutions, either distilled water or 2N NaOH, were then measured (200 g) into the plastic reacting containers. The containers were then put inside their respective Dewar flasks and allowed to come to a stable temperature which was recorded. A software called "Automatic Measurement System" was used to sense the temperatures by the thermistors and record them at fixed intervals. At this stage, the PC was set to record and store the temperature readings. The calorimeters were allowed to record stable temperatures for 40-60 min. before the reactions could be started. The reactions were started from the openings on the top of the calorimeter box, by pushing the plunger tubes down into the liquid in each of the containers. These were done simultaneously, and the temperatures were recorded for 800 min.

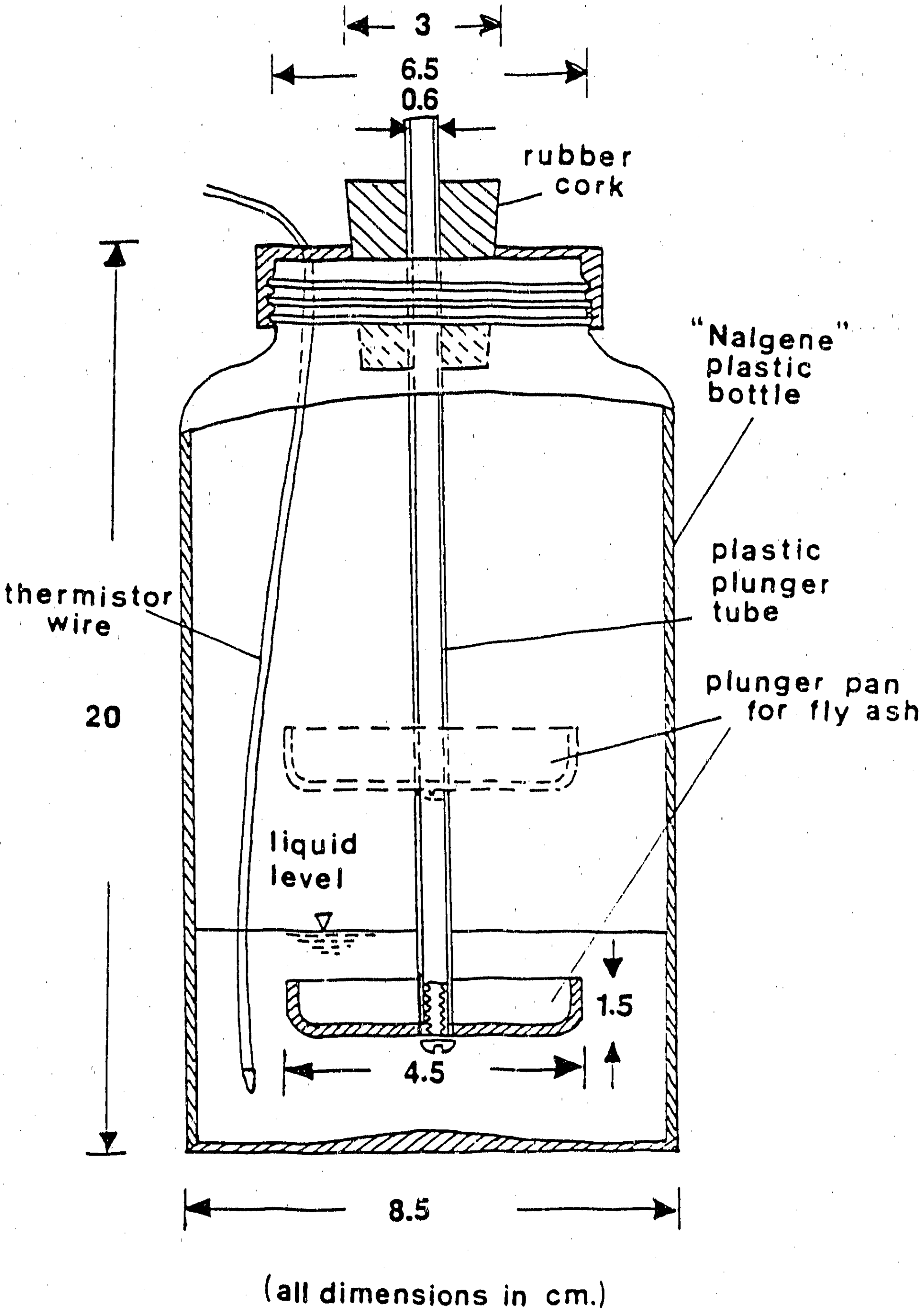

Figure A. The Plastic Reacting Container and the Plastic Plunger Tube-Pan Assembly.

The 2-in. x 4-in. cylindrical samples were cast according to the ASTM C-109 with 20% and 40% of the cement replaced by fly ashes and silica fume. Samples were cast with 7 different fly ashes with a water/(cement + fly ash) ratio of 0.48 which was changed to 0.55 in the case of 40% substitution of cement by silica fume. This was done to achieve equivalent workability with that of fly ash cement pastes. The samples which had 20% of the cement substituted had been cured in a moist room under normal temperature. They were tested in compression after 7 and 180 days, respectively. The samples that were cast with 40% of the cement substituted were cured in a moist room at normal temperature for 14 days and then cured in a water bath at 43°C for 28 days. The elevated temperature was used to enhance the pozzolanic reaction. A set of these samples was cured for 2 days in the moist room at a normal temperature and then cured for 48 hours in the bath at 43°C. The solution in the bath had NaOH, KOH, and $Ca(OH)_2$ in the same proportion as in the pore solution of a cement paste in the long run. Mehta (1984) showed that accelerated curing for 7 days at 43°C produces compressive strength equivalent to that of 28 days. The same is also true for 28 days accelerated curing and 90 days normal curing.

After curing for the required number of days, the samples were tested in compression according to ASTM C109. In the results, the 90-day samples were actually cured for 14 days in a moist room and then cured in a water bath at 43°C for 28 days. The 7-day samples were cured in the moist room for 48 hours and then again for 48 hours in a water bath at 43°C.

Results and Analysis

Table 1. Results of X-ray Analysis.

Elemental Composition (g atoms/100 g of fly ash)

	N#4	CL	OTM	N#2	N#3	AM	WES
Si	0.54	0.597	0.565	0.805	0.638	0.577	0.569
Al	0.396	0.368	0.382	0.323	0.386	0.337	0.353
Fe	0.069	0.069	0.066	0.088	0.124	0.079	0.065
Ca	0.482	0.442	0.453	0.278	0.360	0.426	0.474
Mg	0.119	0.168	0.121	0.060	0.136	0.129	0.139
S	0.036	0.027	0.044	0.021	0.033	0.069	0.033
Na	0.077	0.071	0.100	0.003	0.000	0.074	0.039
K	0.008	0.008	0.008	0.036	0.026	0.013	0.008
RATIOS:							
Al/Si	0.733	0.616	0.676	0.401	0.605	0.584	0.620
ΣZ[a]	1.425	1.437	1.388	0.891	1.266	1.355	1.403
ΣZ/Al	3.59	3.9	3.63	2.76	3.28	4.02	3.97
ΣZ/ (Al+Si)	1.52	1.489	1.465	0.79	1.23	1.482	1.52

[a] ΣZ = ΣNa+K+ 2(Fe+Ca+Mg) g atom/100g (charge equivalents).

Table 2. Compressive Strengths of Fly Ash Mortars, psi.

	N#4	CL	OTM	N#2	N#3	AM	WES
1) 90 days*							
40%	7091	6700	7221	8358	7933	4900	6870
S.D.	734	320	560	250	685	544	360

Control: 6180 ± 200 psi

2) 7 days**							
40%	3978	4246	4549	3928	3874	2371	4339
S.D.	247	702	398	370	129	233	88

Control: 5258 ± 200 psi; silica fume: 5072 ± 287 psi

	N#4	SiF	OTM	LA	N#3	AM	WES
3) 180 days***							
20%	9260	10373	8164	8372	10074	7386	6067

	P.C.	N#2	Otm	Cl	N#3	Am
4) 7 days****						
20%	5668	5448	5632	5790	5703	4650

* Simulated 90 days, (Mehta, 1985) 48 hours air curing at 100% humidity and 22°C, then curing in a water bath at 43°C for 28 days.

** Simulated 7 days, (Mehta, 1985) 48 hours air curing at 100% humidity and 22°C, then curing in a water bath at 43°C for 48 hours.

*** 180 days curing at 100% humidity and 73°C.

**** 7 days curing at 100% humidity and 73°C.

Table 3. Heat Evolution (°C) in the Reaction of 20 g of Fly Ash and 200 g of 2N NaOH.

	N#4	CL	OTM	N#2	N#3	AM	WES
After 100 min.	0.55	0.7	0.45	0.4	0.35	1.00	0.47
After 200 min.	0.6	0.9	0.5	0.4	0.55	1.05	0.6

Table 4. Heat Evolution (°C) in the Hydration of 20 g of Fly Ash and 200 g of Water.

	N#4	CL	OTM	N#2	N#3	AM	WES
After 100 min.	0.7	0.45	0.8	0.1	0.2	1.3	0.8
After 200 min.	0.85	0.5	0.77	0.15	0.3	1.35	0.92

Summary of Findings

1. The reaction taking place in a strongly alkaline environment (pH>13) inside the calorimeter at room temperature is at least a part of the pozzolanic reaction. Generally speaking, it is the breaking down of the amorphous fly ash glass and the consequent formation of hydration products.

2. Reaction and consequent heat generation with strong NaOH and the fly ashes that contain large modifier cations in the glassy phase are vigorous and comparatively quicker than those occurring with the pozzolanic fly ashes containing less modifier cations (Figures 1 and 2).

3. The more modifier cations the amorphous fly ash glass has, the less its silica content, and thus less silica is available in the pozzolanic reaction in the cement fly ash paste. Less silica in the fly ash gives rise to a weaker structure arising from fewer hydration products in the cement fly ash paste compared to when fly ashes with very little modifiers are used as an admixture. Since heat evolution (expressed as temperature rise) in the reaction with NaOH can be used to evaluate a fly ash in terms of modifier cation content of its glassy phase, it can be considered as an indirect way of measuring how ineffective a fly ash can be in terms of long-term strength development in the cement paste (Figure 3).

4. The $\Sigma Z/Al$ ratio, (number of charge equivalents of positive cations in 100 g)/(number of g atoms of Al), is a unique way of measuring the extent of strain in the amorphous glass. The low-modifier fly ash glass has a $\Sigma Z/Al$ ratio around 1, whereas fly ash with high modifier content will have higher ratios (about 4.5). By comparing Figures 4 and 5, it can be noticed that

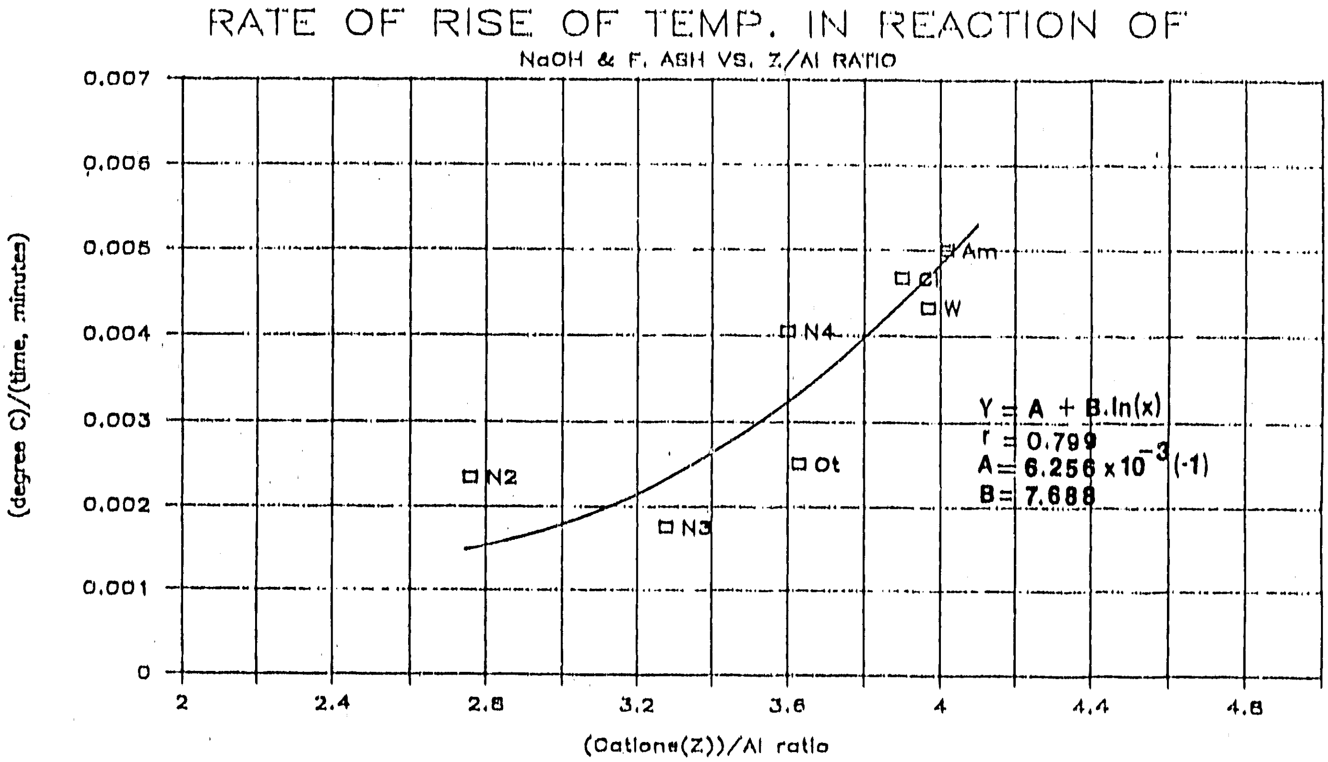

Figure 1. Rate of rise of temperature in the reaction of 200 grams of 2N NaOH and 20 grams of fly ash versus the (Cation (ΣZ))/Al ratio. Cations are expressed in charge equivalents and Al is expressed in gram atoms per 100 grams of a fly ash.

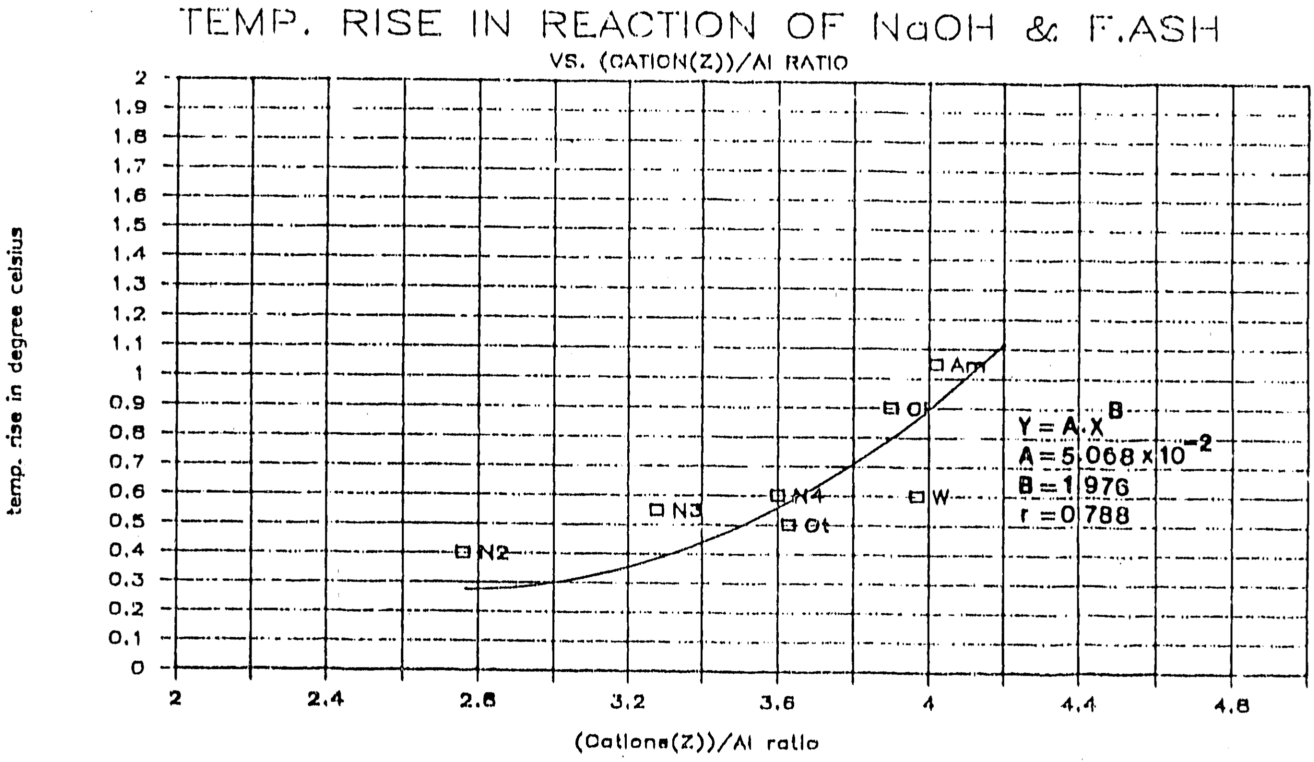

Figure 2. Temperature rise in the reaction of 200 grams of 2N NaOH and 20 grams of fly ash versus the (Cation ΣZ))/Al ratio. Cations are expressed in charge equivalents and Al is expressed in gram atoms per 100 grams of fly ash.

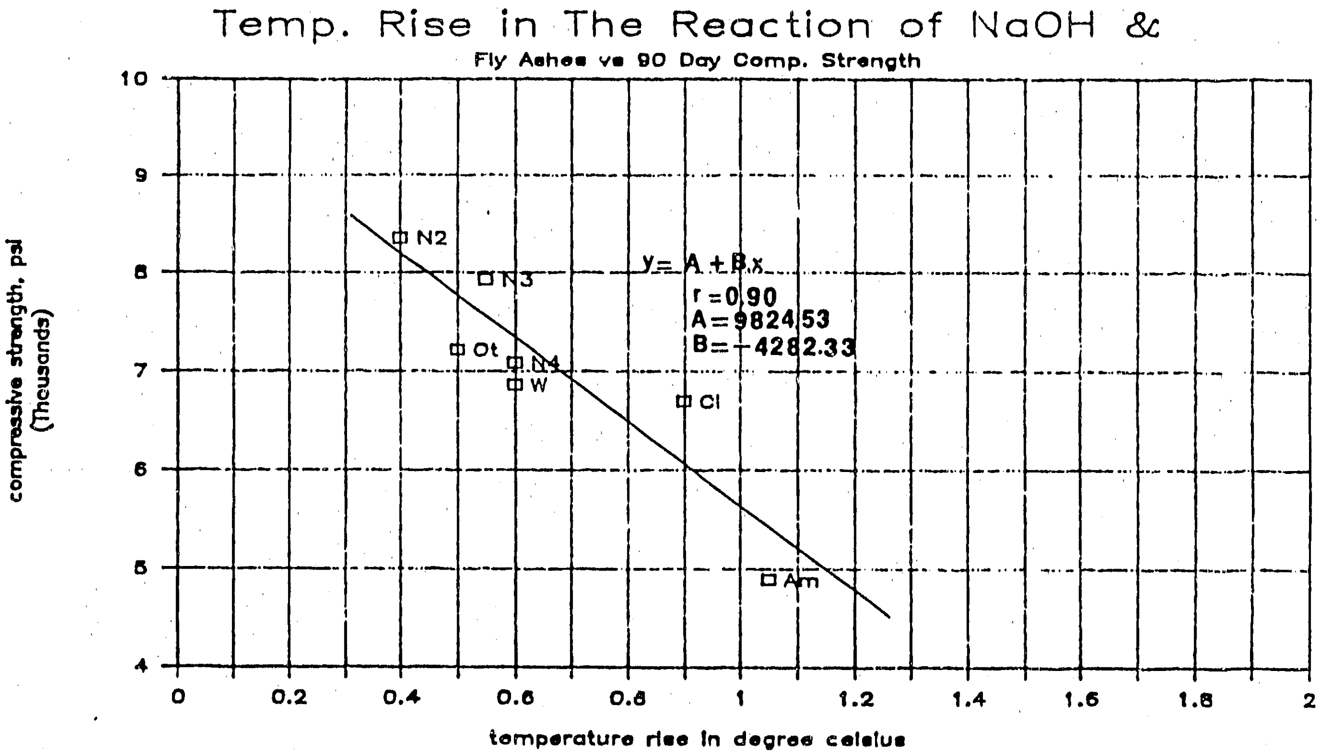

Figure 3. Temperature rise in the reaction between 20 grams of fly ash samples and 200 grams 2N NaOH, against the 90-day compressive strength of 40% fly ash cement mortar.

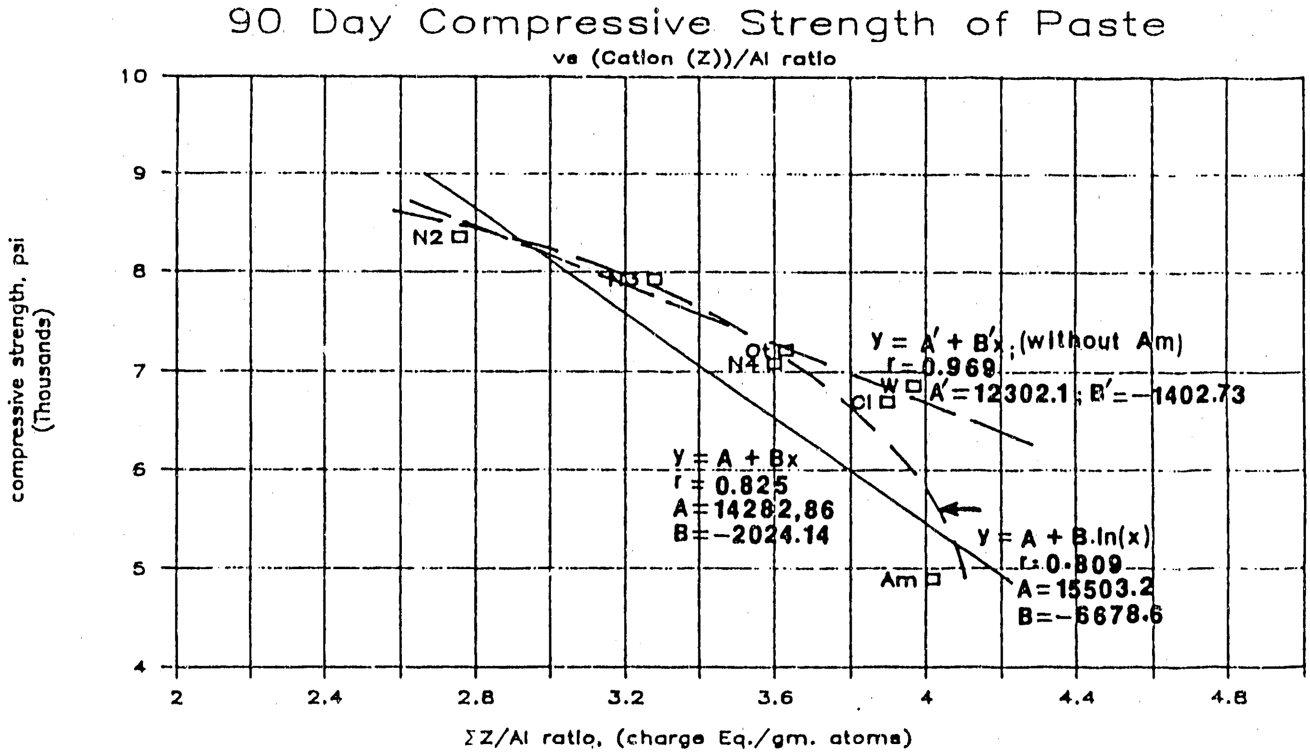

Figure 4. ΣZ/Al ratio of the fly ash samples and the 90-day compressive strength of 40% fly ash cement mortar. The Z is expressed in terms of charge equivalents (Na+K+2(Ca+Mg+Fe)) of the modifier cations and Al is expressed as gram atoms in 100 grams of a fly ash sample.

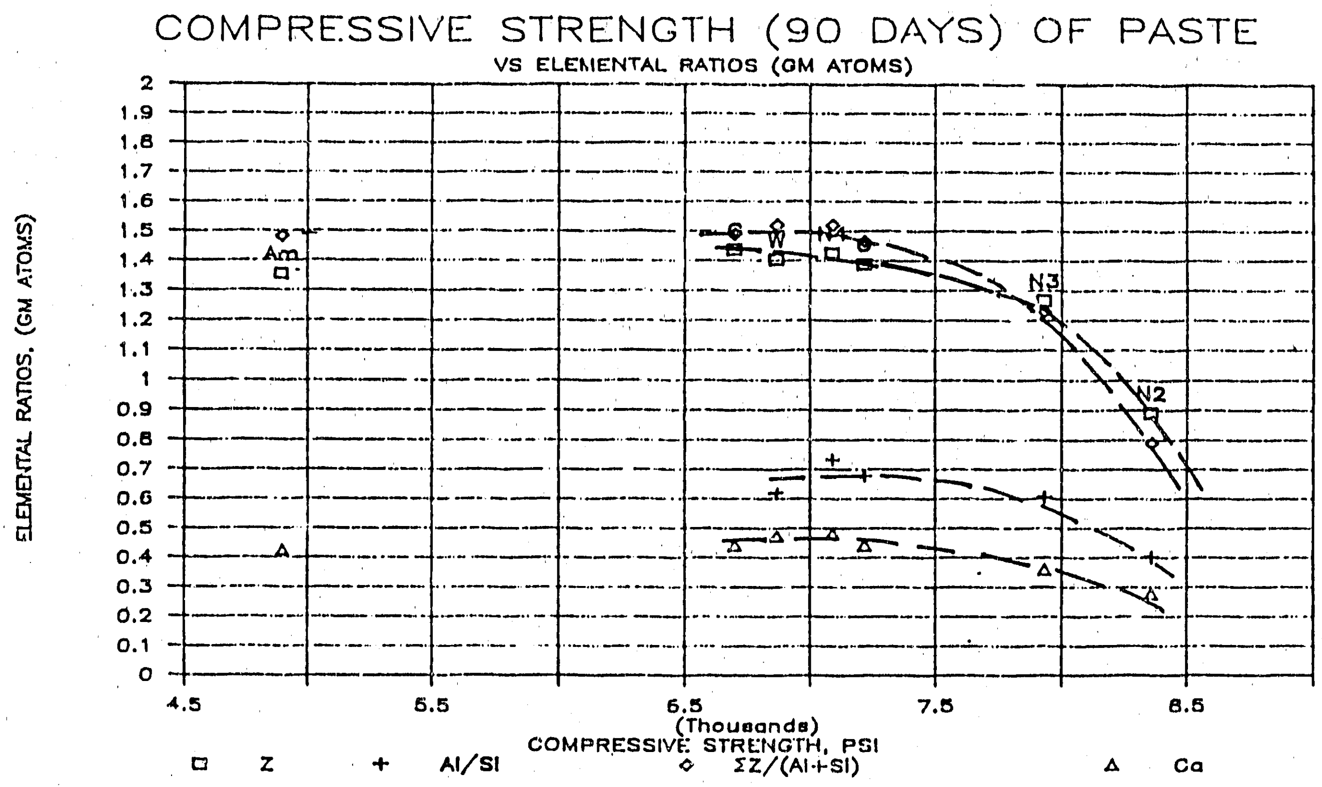

Figure 5. Different elemental ratios and compositions that are used to express the modifier composition, and the 90-day compressive strength of 40% fly ash cement mortar. The different elemental composition and ratios used are Al/Si, ΣZ/(Al+Si), ΣZ and Ca. All the elemental compositions are expressed as gram atoms in 100 grams of fly ash and ΣZ is expressed as charge equivalents of cations in 100 grams of fly ash.

ΣZ/Al ratio has a good relation with the extended compressive strength compared to any other ratios or composition of elements measured to express modifier content or strain in the fly ash glass (e.g., ΣZ/(Al+Si), ΣZ, Ca).

5. From the relationship between the temperature rise in the reaction of 20 g fly ash and 200 g 2N NaOH and the 90-day compressive strength of 40% fly ash cement mortar (Figure 3 and Tables 2 and 3), the fly ashes can be classified into three broad groups based on the temperature rise (in °C): 0-0.5, 0.5-0.9, and 0.9 and above. These groups are I, II and III, respectively. It can be noticed that group III fly ashes will reduce the strength of the fly ash cement mortar compared to the control mix in an extended period, whereas group II will slightly increase or keep the same strength, and group I will increase the strength to a considerable extent (15% or more).

Future Work

Argha Saha will be pursuing his Ph.D. in Civil and Construction Engineering at Iowa State University in the Fall. He will continue to be an ISMMRRI research assistant for 1990-1991 school year.

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Miscellaneous Projects Partially Supported By ISMMRRI

THE THIRD INTERNATIONAL CONFERENCE ON PROCESSING AND UTILIZATION OF HIGH-SULFUR COALS
NOVEMBER 14-17, 1989, AMES, IOWA

Interest in the utilization of high-sulfur coal continues to grow throughout the world as other economical sources of energy are depleted while the demand for energy climbs unabated. But utilizing high-sulfur coal without damaging the environment presents a great challenge. To meet this challenge, countless scientists and engineers in a number of countries are engaged in developing the science and technology needed for utilizing high-sulfur coal in a benign way. Many of these workers were brought together to exchange technical and scientific information at the Third International Conference on Processing and Utilization of High-Sulfur Coals held at Iowa State University on November 14-16, 1989. This conference was part of a series which was initiated at Ohio State University by Professor Yosry A. Attia in 1985. The second conference in this series was held at Southern Illinois University at Carbondale in 1987. It has been proposed that these conferences continue to be held every two years.

The 1989 Conference in Ames, Iowa, was truly international in scope, with papers and information contributed by workers from twelve different countries. The papers provided a summary of the most recent accomplishments by many of the leaders in the field of high-sulfur coal research and development. The papers were selected only after thorough peer review to uphold a high standard of excellence. The carefully edited and revised papers published in this volume represent a report of some of the most comprehensive research and development efforts taking place in industry, government, and academia.

Many of the papers presented at the 1989 Conference dealt with improvements in coal cleaning technology by application of either physical, chemical, or biological processes and various combinations thereof. Included were several papers reporting new developments in froth flotation and oil agglomeration methods. Improvements were noted in coal/pyrite separation by use of chemical and biological surface-modifying agents. Improvements in froth flotation separations were also achieved by application of column flotation or an air-sparged hydrocyclone. A new technique was reported for determining the agglomerability of fine-particle suspensions by monitoring changes in turbidity. Innovative electrostatic and aerodynamic methods were described for beneficiating coal. Since these methods are generally applied to dry, pulverized coal, they could be integrated with the coal-grinding circuits of conventional power stations.

Progress in the development of chemical cleaning technology was reported for caustic leaching, oxidative leaching, and low-temperature pyrolysis methods. Several papers dealt with desulfurization and deashing of various coals by leaching with molten caustics; included were results from a continuous test circuit, as well as results from combustion evaluation of such cleaned coals. Many of the contributions on chemical processing of coal came from overseas. Progress on microbial desulfurization of coal was also reported. Several biological systems were described which showed promise for the removal of organic sulfur, as well as for the reduction of sulfur dioxide from flue gas. In one novel approach, the biological desulfurization was explained in terms of electrochemical properties of pyrite.

A number of papers was concerned with the capture of sulfur oxides either during or following combustion of coal. Several papers dealt with sorbent characterization and the effects of different factors and/or additives on sorbent utilization. Other papers addressed the capture of sulfur oxides in various types of combustion systems, including pulverized coal, slagging, fluidized bed, and magnetohydrodynamic systems. Progress was also reported concerning the development of technology for sorbent injection in flue gas ducts.

Several papers dealt with the utilization of high-sulfur coal in various applications, including power generation and conversion into gaseous and liquid fuels. Included were papers which examined the technical and economic implications of cofiring coal and natural gas in electric utility boilers and of combining a multiple-product coal cleaning facility with conventional and fluidized bed utility boilers. One of the papers described the deleterious effects of pyrite oxidation products, such as would be produced by weathering, on the thermoplasticity of coal. Another paper reported encouraging results achieved with a new hydrodesulfurization catalyst made by supporting zinc chloride on silica gel. Also included was a report on the advantageous use of coal preparation plant waste as an additive in the lime-sinter process for recovering alumina from fly ash.

Underlying the main theme of the conference was a series of papers concerned with the characterization of high-sulfur coals. Several papers addressed modern instrumental techniques, such as automated image analysis, EPR, GC-MS, and EXAFS, for the characterization of pyritic or organic sulfur in raw and processed coals. In one paper, the previously reported removal of organic sulfur from coal by extraction was evaluated by careful analysis. In another, the behavior of coal during flotation was explained in terms of surface free energy. Finally, analyses were reported of high-sulfur coals for recoverable metal values.

Because of the diversity and comprehensive nature of the papers presented at the conference, a book was published based on the proceedings to provide an overview, status report, and in-depth coverage of current research and development work directed towards the processing and utilization of high-sulfur coals. It is a record of what has been accomplished, indicates the direction of current efforts, and suggests needs and problems requiring further work.

THE ORIGIN OF BONDED DEPOSITS IN FLUIDIZED BED BOILERS

Dr. Robert Brown and Balmurli Natrajan
Department of Mechanical Engineering

Objective

The objective of this research is to understand the mechanism by which bonded deposits are formed in the bed of fluidized boilers. These deposits can interfere with boiler operation and have been responsible for unscheduled shutdowns in commercial units. An understanding of these processes will lead to operating strategies to prevent their formation.

Background

Two circulating fluidized bed boilers recently installed at Iowa State University experienced a loss of fluidization on one occasion during the start-up peri-d. A bonded and layered deposit was found to have formed in the lower section of the combustor where a dense phase of bed material exists during combustion operation.

Dawson et al. (1) have analyzed these samples for major, minor, and trace elements using a variety of analytical instruments. They found the deposits to consist of individual particles with layered compositions. The inner core was anhydrite, with outer layers often containing enrichments of iron. On the basis of their results, they hypothesized that locally rich conditions in the bed of the combustor were responsible for deposit formation.

Our work was undertaken to test this hypothesis. Experiments are being performed in a bubbling, fluidized bed combustor located in our laboratory.

Progress

The operation of the bubbling bed combustor was modified to simulate as closely as possible the reaction environment of the circulating boilers. We obtained the same coal and limestone feedstocks as used in the circulating boilers. Provisions were made to add secondary air above the bed to simulate the addition of secondary air in the circulating boilers above the dense bed that is located in the bottom of the combustor. Calculations were completed on primary and secondary air requirements appropriate to simulating stoichiometries existing in the circulating boilers.

The first test performed burned coal in a bed of limestone and sand. Coal was added continuously at the same air-to-fuel ratio for which the circulating bed boiler was designed. We did not add make-up limestone to the bed, although there was enough limestone in the original charge to the bed to remove sulfur for at least two hours of testing. Our goal of this test was to see whether we could induce agglomerate formation in our laboratory combustor.

After about 70 minutes of operation, bed temperature suddenly started to rise above the steady-state level of about 1560°F, followed shortly by a sharp increase in SO_2 emissions. These results are illustrated in Figure 1. Since the temperature rise precedes the SO_2 increase, it appears to be responsible for the sulfur emissions; sulfur capture is reduced sharply above 1550°F, the optimum temperature for sulfur capture (2). We suspected that bed defluidization was responsible for the sudden temperature increase. Examination of bed material confirmed this expectation; large deposits of agglomerated bed material were discovered. We are in the process of analyzing these deposits.

Future Work

The graduate student supported by this project recently resigned his position. Future work is dependent on recruiting a qualified graduate student to continue the research.

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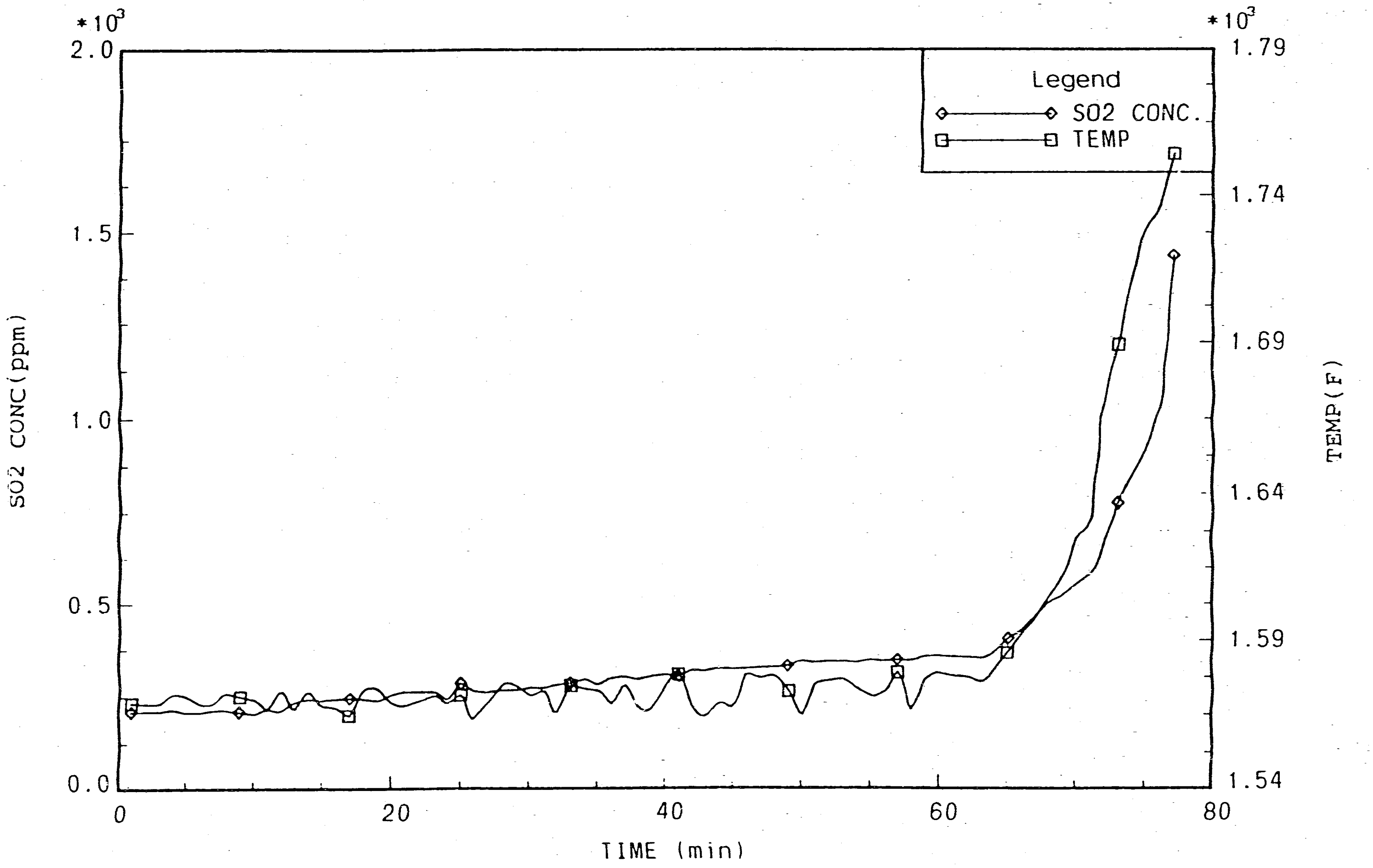

Figure 1. Bed temperature and SO_2 emissions from the combustor vs. time.

SONIC ENHANCEMENT OF FINE COAL CLEANING BY CONVENTIONAL FROTH FLOTATION

William H. Buttermore, Investigator
Iowa State Mining and Mineral Resources Research Institute
and
James Logan, Principal Investigator
Dannette Lowry, Research Assistant
Sonic Resources Corporation, Moline, Illinois

OBJECTIVES

The overall goal of this project is to enhance the effectiveness of fine coal cleaning by conventional froth flotation at finer particle sizes (from 150 to less than 50 micrometers) through the selective application of sonic energy. Efforts involve the demonstration of sonic improvement in coal recovery and quality in laboratory studies using standard coal samples.

Comparisons have been made for laboratory-scale flotation test performed with and without the application of sonic energy for two samples of Illinois Basin coal. Project tasks have included:

1. The design and fabrication of sonic treatment cells.
2. Preparation and characterization of coal samples.
3. Selection of reagent types and concentrations for each coal.
4. Performance of tests to establish optimal sonication parameters.
5. Evaluation of effects of sonication on coal recovery and quality.

INTRODUCTION AND BACKGROUND

Advanced physical coal cleaning processes promise economical, short-term solutions to the environmental problems associated with the combustion of sulfur-bearing coals. Most of these processes exploit the increased liberation of pyrite and ash-forming mineral matter obtained by reducing the particle size of the coal. As particle size decreases, particle surface phenomena overcome forces of gravity commonly exploited for coal and mineral separation. Currently, the most commonly used surface-based separation technique is froth flotation. The effectiveness and economy of this method decrease as particle size decreases. This project seeks to enhance the effectiveness of froth flotation at finer particle sizes through the application of sonic energy.

The successful completion of this project would extend the useful cleaning range for froth flotation and benefit state and national goals of producing clean energy from coal, our most abundant energy resource. Because this research is directed toward enhancing installed capacity for the most prevalent fine-coal cleaning technique, froth flotation, the potential impact is great for short-term benefits in improved capability for the production of cleaner, fine-sized coal.

EXPERIMENTAL PROCEDURES

The approach of this work has been to exploit the dispersive properties of near-field sonic energy to clean particle surfaces, reduce reagent consumption, and extend the useful range of conventional froth flotation from its current lower limit to -325 mesh (44 micrometers) or even smaller. In this way, resulting improvements in technology will have immediate applicability to currently installed capacity.

Two samples of coal, IBC-104 and IBC-106, were obtained from the Illinois coal sample bank, crushed to a nominal top size of 100 mesh (150 μm), and divided by riffling for analysis and laboratory studies. Proximate, particle size, and washability analyses were performed for each sample. An inert storage system was fabricated, and placed in service to minimize the effect of sample aging during the course of laboratory testing. A novel sonic cell was designed and fabricated incorporating one 10-kHz and one 20-kHz sonic transducer on opposite walls of a standard 2-liter Denver flotation vessel. The transducers were powered by a 200-300W variable output generator. A preliminary series of experiments was performed to select reagent type and dosage for each coal. Flotation tests have been performed with and without sonic energy both before and during the flotation procedure.

RESULTS AND DISCUSSION

Results to date indicate that low-power sonication applied as a pretreatment is effective in improving energy recovery and at the same time reducing the product ash content for both sample IBC-104, a difficult-to-clean Illinois No. 6 raw coal containing nearly 40% by wt. ash-forming mineral matter, and sample IBC-106, a washed Springfield coal. Comparing results to reference samples not exposed to sonic energy but treated identically in every other way, overall energy recovery for IBC-104 was improved nearly 5% (from 75.7 to 80.4%), as a result of a 30-second exposure to 200W of 10-kHz sonic energy (Figure 1). For IBC-106, a 5-second exposure to sonic energy slightly improved energy recovery from 87.8 to 88.8%, while reducing the sulfur emission potential in this test by approximately 3%.

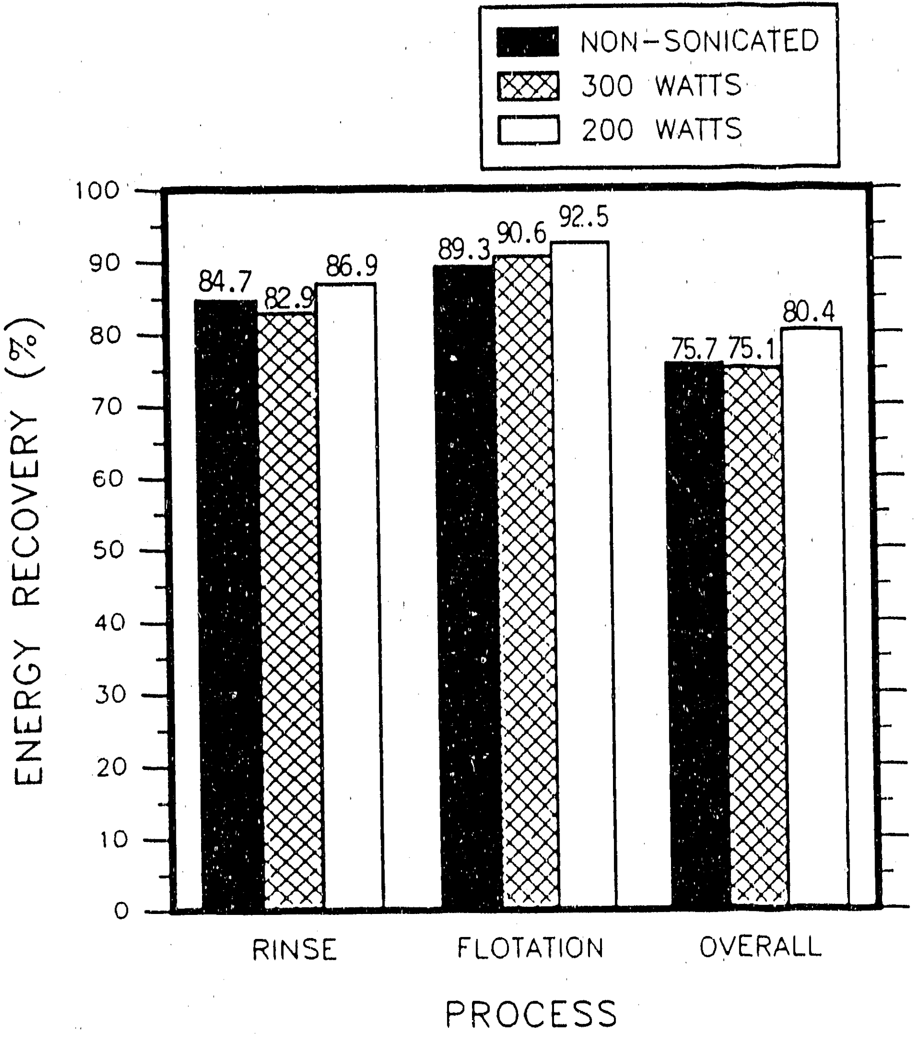

Figure 1. Energy Recovery Comparisons for Non-Sonicated and 30-Second Sonic Pretreatments of Illinois Coal Sample IBC-104.

Although the innovative cell design permits sonication during flotation itself, this research as well as that of an other ISMMRRI project indicate that sonic beneficiation is best realized for Illinois No. 6 coal when samples are exposed to sonic energy and then rinsed thoroughly prior to froth flotation (Figure 2).

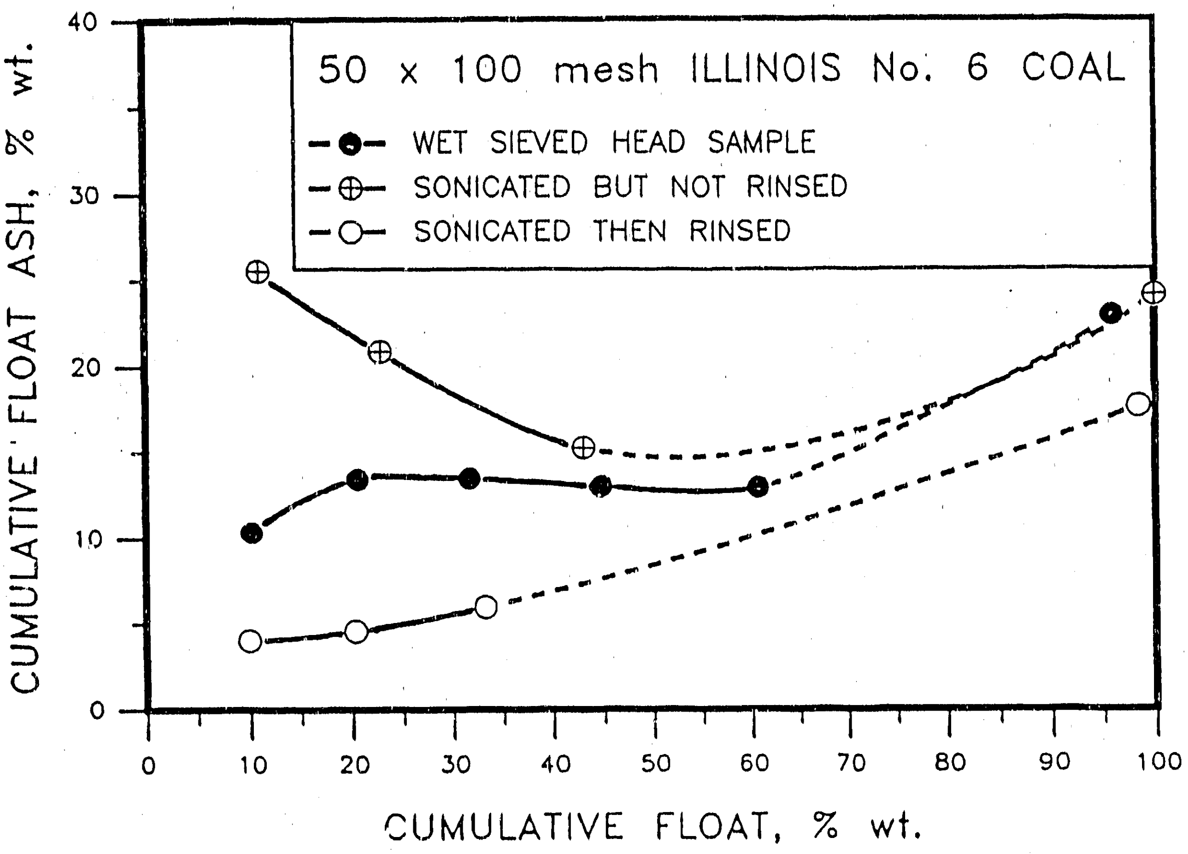

Figure 2. The Effects of Rinsing Following Sonication on Hallimond Tube Flotation of Illinois No. 6 Coal.

Results also suggest that lower power levels and exposure times may improve the beneficial effects of sonication for this coal. Finally, it has been determined from this work that the same sonic treatment method may not be effective for all samples of coal, and that significant improvements in grade and yield can be achieved through specialized application techniques, custom-designed for each individual coal.

THE EFFECTS OF ADSORBED ORGANIC MATTER ON GYPSUM DEHYDRATION

Dr. Robert Cody and Thad Slaughter
Department of Geological and Atmospheric Sciences

Introduction

Little is known about the relationship between adsorbed organic matter and the process of gypsum dehydration. A better understanding of the relationship between gypsum dehydration and adsorbed organic matter could elucidate the role evaporitic sediments play in producing potential source rocks for petroleum generation.

Studies investigating gypsum dehydration reactions, using differential thermal analysis (DTA) and differential scanning calorimetry (DSC), of pure gypsum are numerous (Smykatz-Kloss 1974, 1982; Mackenzie 1970; Todor 1976; Clifton 1971). Heating pure gypsum produces three distinct reactions occurring between temperatures of 25° and 500°C, with three peak temperatures at approximately 150, 200, and 275°C depending on sample preparation techniques. During the heating process, the first 1-1/2 molecules of H_2O are lost and this is represented by the endothermic reaction at 150°C, peak "A". At approximately 200°C, the remaining 1/2 H_2O molecule, represented by peak "B", is lost from $CaSO_4(1/2\ H_2O)$. The third reaction results from the structural change of an anhydrite polymorph. The reaction is exothermic and is attributed to the lattice modification, from a hexagonal to an orthorhombic unit cell, of anhydrite.

DTA has been commonly used for the study of adsorbed organic matter on clay minerals, usually smectites (Yariv et al. 1988; Yariv 1985; Jordon 1949; Grauer et al. 1983; Grauer et al. 1987; Paterson and Swaffield 1980). Heating hydrated, aliphatic, organo-clay complexes, under a flow of dry nitrogen or air, produces thermograms that show no significant change in the shape or signature of the endothermic curve associated with the expulsion of water from the clay interlayers. These studies show a reproducible decrease in the peak temperatures of the dehydration curves during heating of the organo-clay complexes. A relatively large decrease in the peak temperature, during dehydration, is correlated with an increase in the adsorbed organic matter on the internal surface of the clay mineral. This phenomenon is explained by changes in the bonding energies of the water molecules with respect to the adsorbed organic matter and internal clay surfaces.

Objective

The objective of this study was to ascertain the effect of organic matter on gypsum dehydration. Several factors concerning the organo-gypsum samples will be considered in this study: organo-gypsum precipitated in various aqueous systems, organo-gypsum complex morphologies, amount of organic matter adsorbed to gypsum, and type of organic matter.

Procedure

Gypsum precipitated in the presence of organic acids in aqueous systems with ionic strengths, $I = 0.5$, 1.5, and 2.5, provide the organo-gypsum samples used for the DSC analyses. Di- and monocarboxylic, saturated, straight-chain, organic acids used in the study were: stearic (C_{18}), palmitic (C_{16}), myristic (C_{14}), lauric (C_{12}), octanoic (C_8), hexanoic (C_6), hexadecanedioic (C_{16}), 1,12-dodecanedicarboxylic (C_{12}), 1,10-decanedicarboxylic (C_{10}), adipic (C_6), and succinic (C_4) acids.

Precipitating solutions were mixed with various quantities of NaCl: 0.00, 58.44 and 116.88 g/l, to adjust the ionic strength of each solution to 0.5, 1.5, and 2.5, respectively. The pH of all solutions was adjusted to 8.55 with NaOH before initiation of gypsum precipitation. Three different organic acid concentrations of approximately 1.5×10^{-4}M, 6.0×10^{-4}M, and 6.0×10^{-3}M (by volume) of each acid were mixed for 6 days with 50 ml aliquot of 0.5M Na_2SO_4 of varying ionic strength. A 50-ml aliquot of 0.5M $CaCl_2(2H_2O)$ was injected at a rate of 1 ml/min. into the 50 ml aliquot of Na_2SO_4. After gypsum precipitation, the organo-gypsum samples were washed several times to remove trapped organic matter, dried, weighed, and prepared for thermal analysis. Gas chromatography (GC) and Thermogravimetric analysis (TGA) were used to determine the amount of organic matter removed by the precipitating gypsum.

A DuPont 910 Differential Scanning Calorimeter System was used to evaluate the effects organic matter had on the dehydration temperatures of gypsum precipitated under the experimental conditions outlined. Samples were run under dry nitrogen and atmospheric pressure. A heating rate of 5°C/min. was used during the dehydration of the organo-gypsum complexes.

Work Accomplished

The evaluation of DSC data was showed that the heating of di- and monocarboxylic, organo-gypsum complexes produced changes in onset and peak temperatures of the initial dehydration reaction, peak "A", and had no effect on the second dehydration reaction, peak "B", or the third reaction, relative to heated pure gypsum. The following experimental parameters were evaluated with respect to the observed changes in temperature and shape of dehydration reactions associated with di- and monocarboxylic organo-gypsum complexes: 1) ionic strength of precipitating solution, 2) initial organic acid concentration, 3) carbon chain length of organic acid, 4) amount of organic matter adsorbed by precipitating gypsum, and 5) organo-gypsum crystal morphology. Evaluation of organo-gypsum thermal data revealed that three parameters, i.e., gypsum morphology, amount organic matter adsorbed, and carbon chain length of organic acid, were primarily responsible for the observed changes in temperature and shape of the initial dehydration reaction curve of the complexes.

Gypsum precipitated in the presence of di- and monocarboxylic (C_4 - C_{18}) organic acid homologs in various aqueous systems provide the organo-gypsum samples used for DSC analyses. Under the experimental conditions, long-chain organic acids (C_{12} - C_{18}) formed Ca-salts during the gypsum precipitation experiments and adsorbed to the {010} crystal face, producing tabular gypsum habits. Short-chained organic acids (C_4 - C_8) formed free carboxylate anions which adsorbed to crystal faces {110} and {111}, producing prismatic gypsum habits. Adsorption affinity of monocarboxylic acids for the gypsum mineral increased with decreasing carbon chain length of the fatty acid and with a decrease in ionic strength of the saline solution. Adsorption affinity of dicarboxylic acids for the gypsum mineral increased with an increase in the carbon chain length of the acid and with a decrease in ionic strength of solution.

Heating short-chained, organo-gypsum complexes resulted in the reduction of onset and peak temperatures of peak "A", relative to pure gypsum. The greater the amount of organic matter adsorbed, the greater the decrease in the temperatures associated with peak "A". A second reaction was indicated by the presence of a shoulder or secondary peak in the initial endothermic reaction. The additional peak or shoulder represents the endothermic reaction resulting from the desorption of the organic matter from the crystal surface. The desorption of the organic matter is directly related to the initial removal of water from the crystal structure. This phenomenon is independent of heating rate or temperature of dehydration.

Heating long-chained, organo-gypsum complexes resulted in an increase in onset and peak temperatures of peak "A", relative to pure gypsum. The greater the amount of organic matter adsorbed, the greater the increase in the temperatures associated with peak "A". There was no secondary reaction associated with the heating of these complexes. The lack of the second reaction in the DSC analyses of long-chained, organo-gypsum samples is directly related to the site of adsorption of the organic matter. Calcium salts of long-chained, organic acids adsorbed to crystal faces (010), parallel to the water layer; thus when the water is expelled, there is little interaction between the organic matter and the water. This phenomenon is independent of heating rate or temperature of dehydration.

The results of the DSC analyses suggest that the process of gypsum dehydration provides a mechanism for the desorption and transportation of organic matter within a source rock and into a reservoir rock. The data show that organic matter adsorbed to crystal faces subparallel to the water layer in gypsum is desorbed by the process of dehydration. Desorption of organic matter is independent of temperature or rate of heating.

Future Plans

Writing of the Ph.D. thesis needs to be completed. Thad Slaughter has accepted a position as a Geologist with the EPA, Region 5, in Chicago, IL.

Presentations

An abstract based on this work has been submitted for presentation at the Geological Society of America 1990 Annual Meeting, Dallas, Texas, October 29 - November 1, 1990.

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A MINED-LAND RECLAMATION PLAN FOR THE THOMAS HILL ENERGY CENTER IN CLIFTON HILL, MISSOURI

Prof. Norman Dietrich, Ron Spoden and Heidi Williams
Department of Landscape Architecture

BACKGROUND

During the Fall of 1989, Associated Electric Cooperative, Inc. (AECI), agreed to contribute partial support of a graduate research assistant whose master's degree thesis research would be to define a total long-term land management plan for mined-land near the Thomas Hill Energy Center.

The land use and landscape planning of mined-land is a complex process which must synthesize a wide range of often conflicting concerns such as mining productivity, cost-effective reclamation, regulatory requirements, cultural and natural environments, surrounding property, existing land uses and landscape and economic base.

OBJECTIVES

The overall objective of this research project is to examine the opportunities and constraints of this process in conjunction with the operator's goals and objectives for mining, reclamation, and post-mining land use and landscape management, and to give hands-on training in mined-land reclamation. The specific objective is to document the prototypical processes for the application of land-use and landscape planning principles and practices for a large scale surface coal mine (over 24,000 acres) based on the following criteria outlined by AECI:

1. Maximize the benefit/cost ratio.

2. Present a positive image with the community at large.

3. Create a functional and aesthetically acceptable post-mining landscape.

4. Have a positive impact on the social and economic well being of the surrounding area.

5. Be creative and innovative in nature, but at the same time, practical, economical, and easy to implement and maintain.

PROGRESS

During the Spring, 1990, preliminary work was begun on the project. Visits to the Thomas Hill Center were made to ascertain the desires and constraints set by the AECI personnel. The literature and the research work on the Fort Dodge area (a current ISMMRRI-funded project), along with the study of the German Brown Coal District and of the Florida phosphate mining area, were reviewed. Preliminary data gathering (aerial photos, currently proposed land-use maps, property maps, mine plans, etc.) was started by the graduate student Heidi Williams.

Since Ms. Williams has left Iowa State University in late Spring, 1990, the remainder of this project will be completed with the help of another student, Ron Spoden. Additional help will be provided in two courses offered within the Department of Landscape Architecture under the supervision of Professor Dietrich, and in consultation personnel from the Thomas Hill Energy Center in Missouri.

EMPIRICAL MODELING OF AIR-BORNE PARTICLES IN MINING OPERATIONS

Dr. Audrey Levine and Mohamed Abuel-Kassem Mohamed
Department of Civil and Construction Engineering

INTRODUCTION

Control of particles generated during underground and surface mining operations is a safety issue important in mining operations (Tomb et al., 1983). Size distributions of aerosols can be useful for developing dust control strategies to protect workers from potential health effects (Peavy et al., 1985; Vutukuri and Lama, 1986). The health effects caused by inhalation of mine dusts depend upon the contact time, concentration, and chemical composition of air-borne dust particles. Particles that range in size from 0.25 μm to 10 μm are small enough to penetrate into workers' lungs, and therefore, are defined as respirable dust (Marple and Rubow, 1983; Vutukuri and Lama, 1986). Some diseases, such as pneumoconiosis, are caused by inhalation of particles smaller than 5 μm. Inhalation of particles in the size range from 1 μm to 2 μm poses an additional health risk due to lack of mechanisms for removal of these particles by the human respiratory system. In addition, chemical activity tends to increase with decreasing particle size (Hartman et al., 1982; Vutukuri and Lama, 1986).

The major source of mine dusts are the ore being mined. The mining operations responsible for generation of dusts in mine air are drilling, ore cutting, blasting, loading, and dumping. Crushing, grinding, and screening also contribute significant quantities of dust (Drinker and Hatch, 1954; Mohamed, 1985; Vutukuri and Lama, 1986). The chemical composition of dust particles influences the health risk associated with exposure to aerosols.

The acceptable level of dust concentration is defined as the concentration that will produce no adverse health effects. It is specified to be either a fixed value or a function of the chemical composition. When particles contain quartz, the acceptable level of dust concentration is a function of the quartz content of air-borne particles smaller than 5 μm (Vutukuri and Lama, 1986). This relationship is illustrated in Figure 1 where the risk associated with inhalation of respirable quartz is reflected in the magnitude of the allowable dust concentration. As the quartz content increases, the concentration of dust that can be tolerated decreases.

The primary objective of this paper is to report findings from characterization studies of mining particles. Particle size distributions and elemental composition are investigated. Based on the results, the need for control measures within mines and in the vicinity of the mines and quarries can be determined.

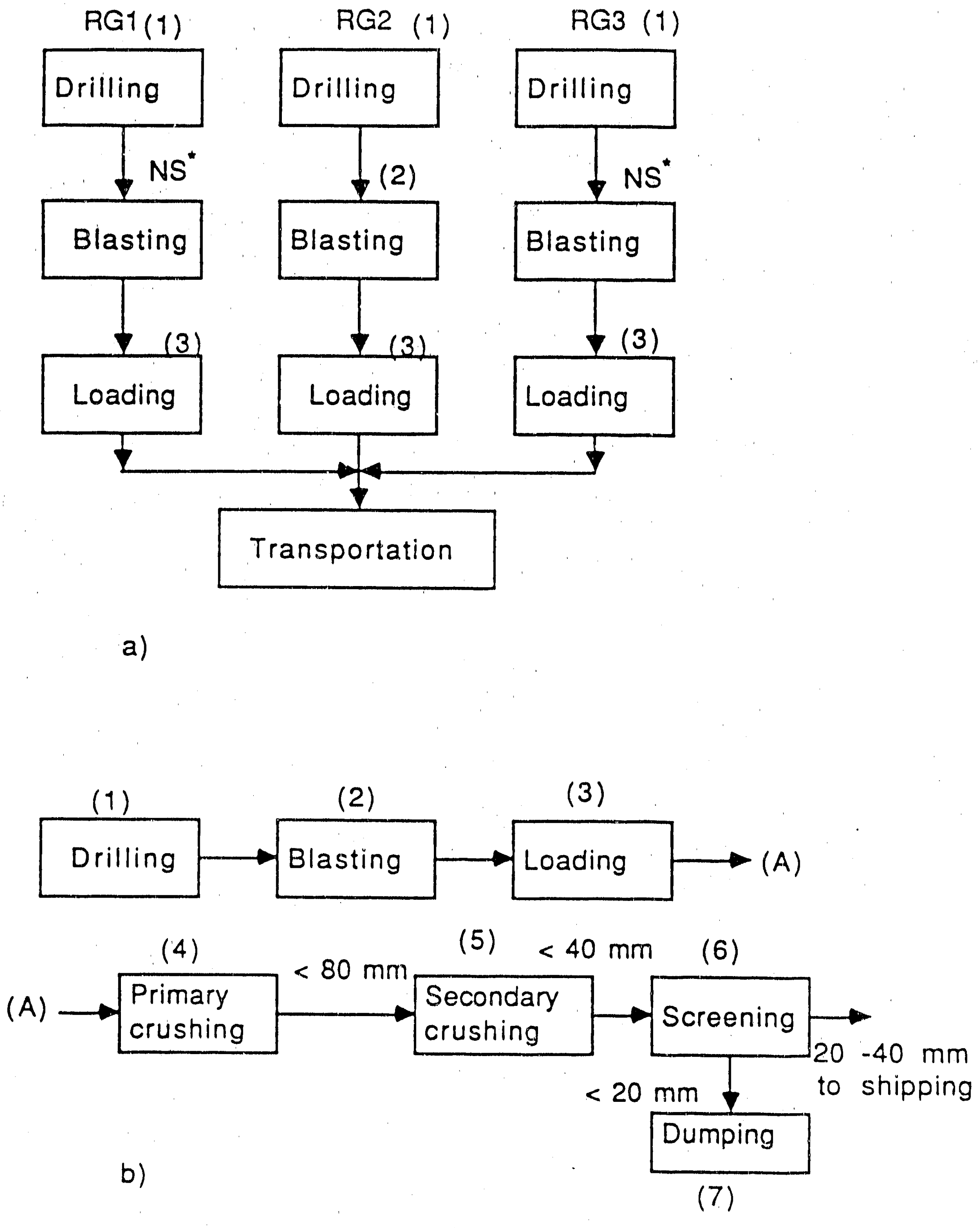


Figure 1. Flow diagram for mining operations, phosphate mine, limestone quarry.

Experimental Methodology

Mine air was sampled at various distances from working faces at two mines in Egypt (due to the difficulty in obtaining access to U. S. mines): 1) a phosphate mine, and 2) a limestone quarry. The sample collection program for the operations at each mine is shown in Figure 1.

Dust samples were collected using a suction pump to aspirate dust-laden air through membrane filters 5 cm in diameter (nominal pore size of 0.8 μm). The flowrate of the suction pump was maintained constant at 0.03 m^3/min.

Particle size distributions were determined for samples of particles collected from operation, and the elemental composition of particles was evaluated. Sample preparation techniques and instrument specifications used for analyzing air-borne particles are summarized in Table 1. Representative particles from each sample were analyzed using scanning electron microscopy coupled with image analysis to evaluate particle size in terms of volume equivalent diameter, length, and width. A LeMont Image Analyzer was used to determine particle size characterization of representative particles. Also, a Kevex microanalyzer was used for elemental composition of analysis.

Table 1. Sample Preparation, Instrument Specification and Operating Conditions for Analysis of Dust Particles.

Item	Description
Sample collection	Dust samples were collected on cellulose acetate membrane filters 5 cm in diameter (nominal pore size of 0.8 μm).
Sample handling	Filters were weighed before and after sampling.
Sample preparation	Small sections were cut from the centers of the filter paper and mounted on carbon scanning electron microscope (SEM) using carbon paint. The mounted filter sections were coated with a thin layer of evaporated carbon film (0.03 μm)
Type of instrument	Scanning electron microscope, made by JEOL (Japan Electron Optics Laboratory), Model JSM840.
Operating Conditions	Magnification 2000x Current 300 picoampere Working distance 39 mm
Number of particles analyzed per sample	From 20 to 68 particles analyzed for elemental analysis and from 176 to 454 particles analyzed for size analysis.
Size range of particles	From 0.007 μm to 39.2 μm.

RESULTS AND DISCUSSION

Air-borne particles generated during mining operations were characterized. Particle size distributions were evaluated, and the chemical composition of air-borne particles generated at each type of mine was estimated.

Particle size and shape were evaluated using scanning electron microscopy. The air-borne particles are small enough to be in the respirable range. The particles have irregular edges that influence transport in the atmosphere and in the human respiratory system.

The effect of mining operations upon particle size is shown in Figure 2. For all operations, the average equivalent diameter of air-borne particles is less than 1.5 μm at the phosphate mine and less than 2.0 μm at the limestone quarry. Thus, mine dust particles are well within the respirable range.

The elemental compositions for air-borne particles collected at the phosphate mine and the limestone quarry are shown in Figure 3 and Table 2. When quartz is present in respirable particles smaller than 5 μm, it poses a greater danger to health than other minerals (Hartman et al., 1982). The allowable dust concentration based on quartz content can be estimated using Equation 5 (Vutukuri and Lama, 1986):

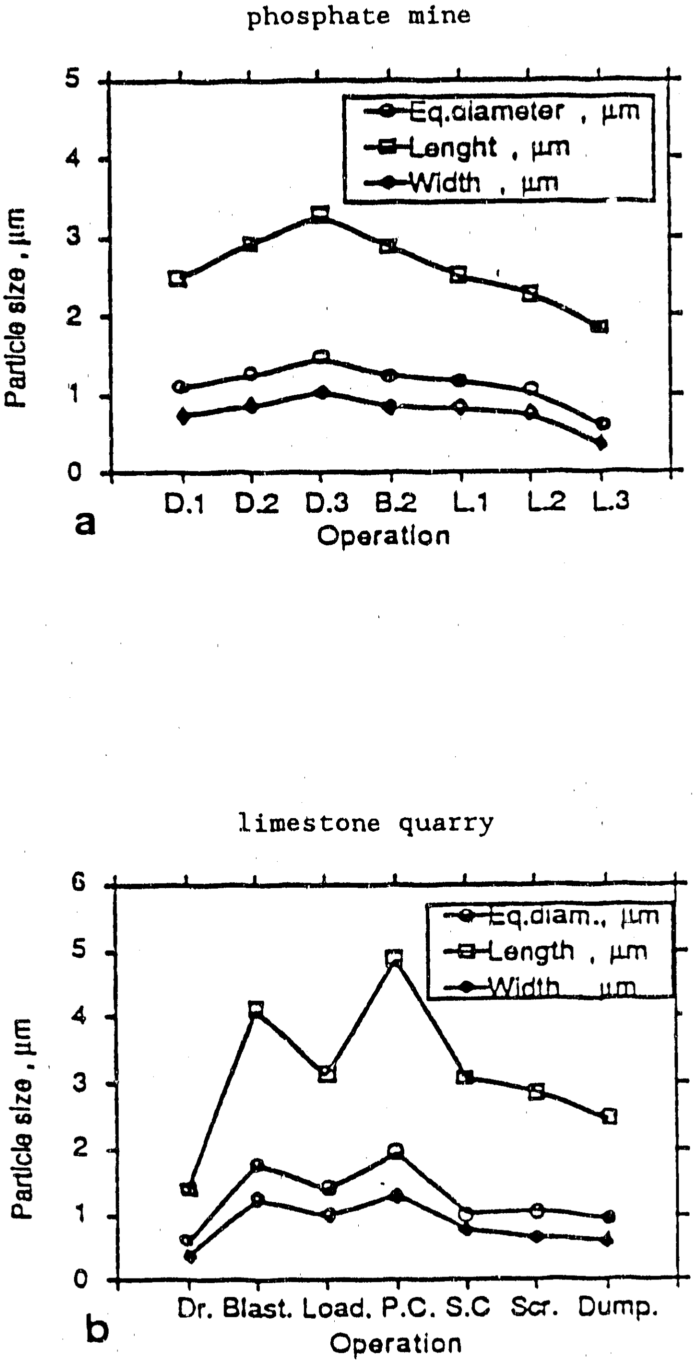
$$\text{Acceptable dust concentration} = \frac{10}{(\%\ \text{air-borne quartz particles} + 2)}$$

Thus, based on quartz content, the dust concentration that can be tolerated without health risks during mining operations at the phosphate mine is 3 mg/m^3. At the limestone quarry, the quartz content is lower and therefore, a dust concentration of 4 mg/m^3 would be acceptable (considering size and elemental composition of dust).

Table 2. Elemental Composition of Air-Borne Particles Collected During Mining Operations at Phosphate Mine and Limestone Quarry.

Chemical Compounds	Composition	Percent of Total Air-Borne Particle Mass	
		Phosphate Mine (s.d.)*	Limestone quarry (s.d.)*
Calcite	$CaCO_3$		87.12 (16.53)
Ca-phosphate	$Ca_3(PO_4)_2$	32.82 (29.94)	0.30 (0.26)
Clay	$Al_2Si_2O_2(OH)_4$		11.76 (0.00)
Fe-compounds	$FeCO_3$, FeS_2 $Fe(SO_4)$	11.90 (22.19)	
Gypsum	$CaSO_4 \cdot 2H_2O$	3.33 (3.46)	0.35 (0.44)
Quartz	SiO_2	1.36 (1.99)	0.70 (0.00)
Silicates	M_2SiO_3	9.00 (8.47)	

* Standard deviation.



D.1 is drilling in RG1
D.2 is drilling in RG2
D.3 is drilling in RG3
B.2 is blasting in RG2
L.1 is loading in RG1
L.2 is loading in RG2
L.3 is loading in RG3

Dr is drilling
Blast. is blasting
Load. is loading
P.C is primary crushing
S.C. is secondary crushing
Scr. is screening
Dump. is dumping

Figure 2. Average particle size in air associated with mining operations, a) phosphate mine, and b) limestone quarry.

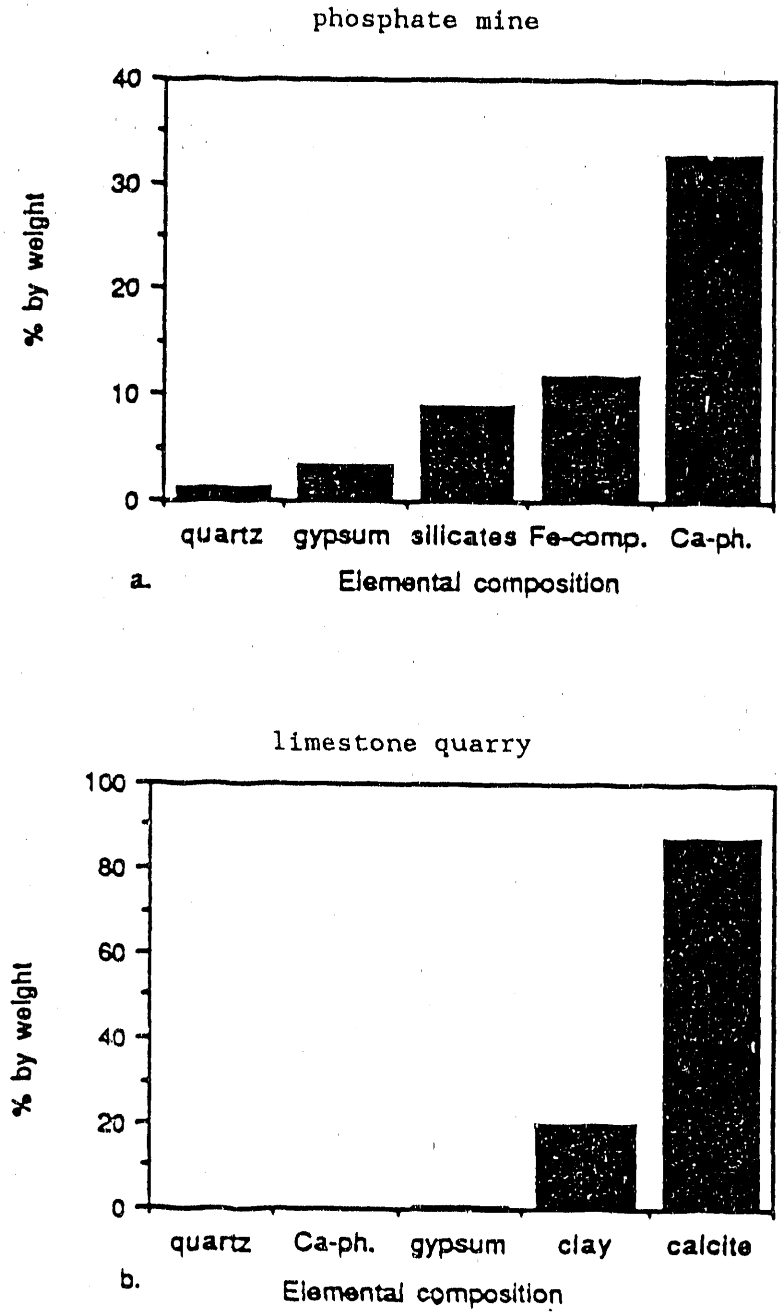


Figure 3. Elemental composition of air-borne particles at the investigated sites, a) phosphate mine, and b) limestone quarry.

CONCLUSIONS

The principal conclusions from this study of air-borne particles at the phosphate mine and the limestone quarry are:

1) The majority of air-borne particles collected from mine atmospheres under all operations were smaller than 5 μm in size, and thus could pose health risks to mine workers.

2) Elements associated with the underground phosphate mine are quartz, gypsum, silicates, Fe-compounds, and Ca-phosphate; those associated with the limestone quarry are quartz, Ca-phosphate, gypsum, clay, and calcite.

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COAL HANDLING CHARACTERISTICS

Dr. Robert Lohnes and Steven Levorson
Department of Civil and Construction Engineering

Objectives

The primary objective of this research, funded by the Electric Power Research Institute and CQ, Inc., is to develop a coal handleability index that will allow utility companies to predict the ease with which coal will move through their systems. Such an index can be used in utilities' coal specifications. Another closely related objective is to develop test methods and hardware that utilities can use to evaluate the acceptability of a coal shipment. A third objective of this study is to establish fundamental relationships between handling characteristics and properties such as ash content, moisture content, hardness, and particle size distribution.

Approach

The primary method of evaluation of the various coals is triaxial testing. Although the method is well known in soil mechanics, it is relatively new to bulk solid handling applications. Direct shear testing of the coals on various hopper materials provides data on wall friction.

As a first approximation, the ratio of unconfined yield strength to bulk unit weight has been selected as a handleability index. The rationale for using this value is that it is a part of the relationship used in calculating minimum hopper openings. Another component under consideration for the handleability index is the wall friction, ϕ'. For a complete analysis to ensure mass flow, ϕ' should be included; however, this parameter is a function of hopper-wall material interacting with the coal, and to be accurately used, it must be measured with the hopper wall material at a specific plant. The final criterion for selecting the handleability index will be its ability to predict the coal's performance in the plant. It is expected that the handleability index will be adjusted as data on each coal's handling characteristics are obtained from operators.

Progress

Several interesting observations and some tentative conclusions have been made. In general, little difference exists in the gradations of the coals studied thus far. The average specific gravities of the coals are about the same ranging from about 1.3 to 1.5.

The cohesions of normally consolidated coals are extremely low. The effective friction angles are nearly the same for all samples tested ranged from about 36° to 39°C. Correlation coefficients in all cases exceeded 0.999.

Although the unit weights of the various coals have a fairly narrow range of about 7.8 kN/m^3 to 8.7 kN/m^3, the unconfined yield strengths vary from zero to almost 4 kPa at 50 kPa consolidating stress. As expected, consolidating stress has the effect of greatly increasing unconfined strength. Increasing moisture contents also appear to increase unconfined strength. The ratio of unconfined strength to unit weight shows a variation from zero to 0.5 m.

Wall friction angles vary from about 16° to 22°, with stainless steel consistently having lower angles. Also, higher moisture contents seem to increase wall friction. The correlation coefficients are all greater than 0.97.

The number of coals studied to date is small and the particle size distribution similar in most cases. Also, the variation in moisture content has been kept within the range of contents expected in normal plant operation. These preliminary results suggest that unconfined strength is a better parameter than effective friction angle to differentiate between coals and that the ratio of unconfined strength to bulk unit weight, i.e., approximate hopper dimension shows promise as an estimator of coal handling problems.

CHARACTERIZATION OF MINERALS AND SULFUR FORMS IN LIGNITES

Dr. Warren E. Straszheim
and Mark A. Nuetzmann and Glenn M. Oren
Iowa State Mining and Mineral Resources Research Institute
and
Department of Civil and Construction Engineering

In 1989, a collaborative effort was begun with Minnesota Power and Light to determine the cleanability of North Dakota lignites. Lignites typically do not require cleaning since they tend to be naturally low in ash and sulfur. However, Minnesota Power had concerns about some samples with sulfur contents high enough that they would not comply with New Source Performance Standards (NSPS) without at least some cleaning. Arrangements were made to use automated image analysis (AIA) and electron beam methodologies developed in Ames to determine the association of the mineral matter (especially pyrite) with the coal, and thus to provide an indication of cleanability.

Subsamples of ground coal were embedded and polished for scanning electron microscopy (SEM) analysis. Organic sulfur contents were directly determined using a microanalysis technique refined in Ames. The difference between total sulfur (determined by ASTM procedures) and the direct organic sulfur determination indicated that the coals had significant amounts of sulfur present as pyrite which should be amenable to removal by physical means. Automated analyses of the coal-mineral matter associations quantified the relationship of coal and minerals in terms of the amount and type of mineral matter in individual particles. Those results showed that the pyrite was especially well liberated, and that it should be subject to removal by conventional physical means.

To confirm SEM analyses, samples of 8-mesh and 50-mesh coals were cleaned by float-sink separations in 1.6 sp. gr. liquids (halogenated hydrocarbons). For both sizes, upwards of 80% of the pyritic sulfur was removed while maintaining more than 97% recovery of the coal (by weight). It remains to be determined what are the optimum, i.e., most economical, conditions and methods for cleaning. However, these lower-rank coals do appear to be very cleanable, and further tests are being planned to explore this possibility.

SECTION III

EDUCATION AND TRAINING PROGRAM

SECTION III
EDUCATION AND TRAINING PROGRAM

The ISMMRRI interdepartmental education and training program exposes students to the broad interdisciplinary research directives of the nationwide Mineral Institute program. In addition to the training they receive from their respective academic departments, the graduate students working on projects funded by the Allotment Grant are trained in other disciplines of mining and mineral resources. The faculty in the Institute and at Iowa State University are committed to producing well-rounded scientist and engineers who will become leaders in industry, government, and academia.

Interdisciplinary Minor in Mineral Resources

An interdisciplinary minor in Mineral Resources, proposed by ISMMRRI and established by the Graduate College, is significant to the Institute's education and training program and continues to grow and strengthen with each Allotment Grant. The minor has a total of 19 participating departments from four colleges (Engineering, Agriculture, Science and Humanities, and Design) which are involved in the education and training of graduate students.

1989/1990 School Year
Participating Departments

Agricultural Engineering
Agronomy
Animal Ecology
Chemical Engineering
Chemistry
Civil & Construction Engineering
Community and Regional Planning
Economics
Engineering Science and Mechanics
Forestry
Geological and Atmospheric Sciences
Indust. & Mfg. Systems Engineering
Landscape Architecture
Materials Science and Engineering
Mechanical Engineering
Physics
Nuclear Engineering
Political Science
Sociology and Anthropology

Cooperating departments offer graduate study and research opportunities in areas such as mineral and coal processing, extractive metallurgy, coal utilization and combustion, resource recovery from fly ash, rock mechanics, development of mining equipment, geology of coal and ore deposits, economics of coal mining and transportation, environmental impact of mining, reclamation of surface-mined land, and utilization and containment of wastes associated with mining and mineral processing. The program has helped to tie together various disciplines needed to cope with the complex problems of extracting, processing, and utilizing fossil fuels and minerals, while at the same time maintaining or improving the ecosystem.

The interdepartmental minor in Mineral Resources complements the research activity which is supported by the Institute and has expanded educational opportunities for graduate students interested in the field of mining and mineral resources. The objectives of the minor have been to provide students with a basic understanding of the nature of mineral resources, mining methods, or mineral processing techniques, and of related social, legal, economic, and environmental aspects of mining and mineral extraction operations. The minor has also provided students with training in the application of the techniques of their major field to the solution of coal and mineral resource problems. Thus, the Institute was able to provide significant support for graduate student research and training with funds received from the U. S. Department of Interior. As a testament to this support, ISMMRRI will have graduated 49 Masters and 14 Ph.D. candidates by the end of 1990.

The minor in Mineral Resources is currently pursued by students from the 19 academic departments listed on the previous page. Several of these departments have actively supported the establishment of several new graduate courses and research projects in the area of mining and mineral resources, and provided faculty for teaching and research supervision.

Approved Current Curriculum for Academic Year 1990-1991

The design of the Mineral Resources minor has evolved over the past few years as experience is gained in application of the set goals. The following series of Mineral Resources core courses is currently approved by the Advisory Committee:

Core Courses

Geol.	505	Geology and Mineral Resources	(3 cr.)
C.E.	506	Mining Methods and Operations	(2 cr.)
Geol.	507	Mineral Resources Field Trip	(1 cr.)
Ch.E.	508	Mineral Processing Operations	(3 cr.)
L.A.	509	Mining Reclamation and Mitigation	(3 cr.)

The requirements for a Masters degree include 6 credits or more of courses consisting of the field trip (Geol. 507), plus at least one additional mineral resources core course. The requirements for a Ph.D. degree include 12 credits or more of approved courses, including Geol. 507, and at least two additional mineral resources core courses. The following supporting elective courses may also be included in the minor:

Supporting Courses

C.E.	509	Rock Engineering	(3 cr.)
M.S.E.	514	Advanced Extractive Metallurgy	(2 cr.)
Ch.E.	515	Coal Science and Technology	(3 cr.)
Ag.E.	523	Erosion and Sediment Transport	(3 cr.)
Geol.	543	Microanalysis of Geologic Minerals	(2 cr.)
E.S.M.	560	SEM Characterization of Materials	(3 cr.)
Chem.	576	Surface Chemistry	(3 cr.)
Geol.	582	Economic Geology	(3 cr.)
C.E.	583	Advanced Geomaterials Lab.	(3 cr.)
Min.Res.	590	Special Topics on Mineral Resources	(Var.)
Geol.	682	Seminars in Economic Geology	(1-3 cr.)
IMST	443	Material Handling	(3 cr.)

In selecting courses for the minor, the courses must be selected so as not to duplicate topics or areas covered by other courses taken for the major. Other requirements for the Mineral Resources minor include the preparation of a thesis in the major field which deals with a coal or mineral resources problem. The minor should be developed in consultation with a member of the Mineral Resources Advisory Committee who will also serve on the student's program of study committee. This member should be from an academic discipline outside the major field.

Students Enrolled

During the academic year 1989-1990, and by the spring of 1990, the three doctoral and 14 masters students listed in Table 1 had declared their intentions of minoring in Mineral Resources. These students are associated with six different academic departments and are/were under the direction of 9 different major professors.

Table 1. Graduate Students with a Declared Minor in Mineral Resources for Academic Year 1989-1990 and as of Fall, 1990.

Name	Department	Degree Sought	Major Professor
Dykstra, J.	Mechanical Engineering	M.S./Ph.D.	Brown
Fuhrmann, G.	Geol. & Atmospheric Sciences	M.S.	Spry
Green, T.	Materials Science & Engr.	Ph.D.	Akinc
Lessa, A.*	Chemical Engineering	M.S.	Wheelock
Levorson, S.*	Civil & Construction Engr.	M.S.	Lohnes
Liu, P.	Geol. & Atmospheric Sciences	M.S.	Spry
Merchant, T.*	Civil & Construction Engr.	M.S.	Pitt
McGowan, K.	Geol. & Atmospheric Sciences	Ph.D.	Spry
Natrajan, B.	Mechanical Engineering	M.S.	Brown
Norton, G.	Civil & Construction Engr.	M.S.	Levine
Qiu, X.	Chemical Engineering	M.S.	Wheelock
Saha, A.	Civil & Constr. Engr.	M.S./Ph.D.	Pitt
Spoden, L.	Landscape Architecture	M.L.A.	Dietrich
Tucholski, D.	Mechanical Engineering	M.S.	Colver
Vu, J.	Civil & Constr. Engr.	M.S.	Pitt
Williams, H.	Landscape Architecture	M.L.A.	Dietrich
Zhang, X.	Geol. & Atmospheric Sciences	Ph.D.	Spry

* ISMMRRI assistantships begin Fall, 1990, on new ISMMRRI grant.

Field Trip

During the first week of May, Mineral Resources Course 507 convened for the purpose of a two-day field trip. The first day of the trip, the group had a guided tour of the underground coal mining operation and the rehabilitation performed by the Star Coal Company of Lovilia, Iowa. This was followed by a visit to the Jude Coal Company's surface strip mine, near Bussey, Iowa. Their rehabilitation program was in full operation. The day concluded with tours of the Scott Mine reclamation site, located in Mahaska County, Iowa, as well as the Highway 92 reclamation project. At the Scott site, the students were able to talk to the land owner, Oren Scott, and his son and ask questions about the productivity of the farm land and their feelings on the success of the strip mine operation before reclamation.

Second-day activities included a visit to the U.S. Gypsum Corporation of Fort Dodge, Iowa. There, the students observed a working gypsum strip mine, rehabilitation operations, and wallboard manufacturing operations. During the afternoon, employees of the Martin-Marietta Corporation, also of Fort Dodge, led a tour of their underground limestone mine and the associated limestone processing.

Student Support

A number of assistantships were provided for students to engage in research related to mining and mineral resources. In addition to ISMMRRI, these assistantships whose efforts are specially documented in Section II of this report, additional funds for some research support were also provided by grants and contracts channeled through various administrative units such as the Engineering Research Institute, the Institute for Physical Research and Technology, and the Ames Laboratory.

Career Decisions by Recent Graduates

Career decisions made by students who have been supported with ISMMRRI assistantships in the past are indicated in Table 4. All of the students conducted research in Mineral Resources and the majority enrolled in one or more courses in this area.

Table 4. Known Career Decisions of ISMMRRI Students Graduating Since 1987.

Student	Graduation Date and Department	Career Decisions
Brett Bradfield	Spring '89 (M.S.) Civil Engr.	Civil Engineer Terracon Consultants Kansas City, MO
Robert Carnazzo	Fall '89 (M.S.) Civil Engr.	Technical Representative Asphalt Paving Association of Iowa Des Moines, IA

(continued)

Table 4. Known Career Decisions of ISMMRRI Students Graduating Since 1987.

Student	Graduate Date and Department	Career Decisions
Arnold Doden	Spring '88 (M.S.) Geology	Graduate Student (Ph.D.) Department of Geosciences Pennsylvania State University State College, PA
Jeff Dykstra	Summer '90 (M.S.) Mechanical Eng.	Graduate Student (Ph.D) Department of Mechanical Eng. Iowa State University Ames, IA
Kent Floy	Summer '87 (M.S.) Chemical Engr.	Process Design and Development Engineer Cargill, Inc. Memphis, TN
Hilary Inyang	Spring '89 (Ph.D.) Civil Engr.	Assistant Professor Department of Civil Engineering University of Wisconsin Platteville, WI
Mark Koellner	Fall '87 (M.S.) Geology	Geologist Tethys Consulting Harrisburg, PA
Matthew Kramer	Summer '88 (Ph.D.) Geology	Associate Scientist Metallurgy and Ceramics Program Ames Laboratory Ames, IA
Keith Kutz	Fall '87 (M.S.) Geology	Geologist VPI Blacksburg, VA
Stephen Megivern	Fall '88 (M.S.) Civil Engr.	Civil Engineer Terracon Consultants Witchita, KS
Glenn Norton	Spring '90 (M. S.) Civil & Const. Eng.	Associate Chemist Ames Laboratory Ames, IA
W. Mark Nutt	Spring '89 (M.S.) Nuclear Engr.	Engineer Florida Power & Light Co. Miami, FL

(continued)

Table 4. Known Career Decisions of ISMMRRI Students Graduating Since 1987.

Student	Graduate Date and Department	Career Decisions
Mary Kay Rogge	Summer '89 (M.S.) Geol. & Atmos. Sci.	Environmental Specialist Dept. of Natural Resources Des Moines, IA
Argha Saha	Summer '90 (M.S.) Civil & Const. Eng.	Graduate Student (Ph.D.) Dept. of Civil & Const. Eng. Iowa State University Ames, IA
Thad Slaughter	Summer '90 (Ph.D) Geol. & Atmos. Sci.	Geologist EPA Chicago, IL
John Vu	Fall '90 (M.S.) Civ. & Constr. Engr.	Civil Engineer Iowa Dept. of Transporation Ames, IA
J. David Wonder	Summer '87 (M.S.) Geology	Hydrogeologist Hickok & Associates Des Moines, IA

Seminar Speakers

The Institute-sponsored seminars with invited speakers from industry, government, and academia provide opportunities for ISMMRRI researchers to be updated on new technologies and exposed to real-life problems. Invited seminar speakers and their topics during the past year included:

"Surface Mining and Hydrogeology"
Dr. Christopher C. Mathewson
Texas A & M University
College Station, Texas

"Inorganic Transformations and Ash Depositions During Coal Combustion"
Dr. Steven A. Benson
University of North Dakota
Grand Forks, North Dakota

"Contact Angles and the Oil Agglomeration of Coal"
Dr. Robert J. Good
State University of New York
Buffalo, New York

"Combustion Research at Consolidated Coal Company"
Dr. Dee P. Rees
Consolidated Coal Company
Library, PA

"Statistical Sampling for Chemical Analysis: The Visman Equation and Other Approaches"
Dr. Byron Kratochvil
University of Alberta
Edmonton, Alberta, Canada

SECTION IV

RESOURCES AND RESEARCH INTERACTIONS

SECTION IV
RESOURCES

One of the major goals of ISMMRRI is to foster cooperation, research, and technology transfer among researchers, government and industry. To achieve this goal, ISMMRRI actively interacts with various entities to assist them with problems that have developed, supplies research results, and disseminate mining- and mineral-related information. To this end, ISMMRRI has communicated and actively participated with the agencies and corporations shown on pages 121-123.

The mineral resources research effort at Iowa State University is diverse. This research is performed in university research facilities on and off campus, as described below.

The excellent research facilities of the participating departments in the ISMMRRI program offer an extensive array of laboratories and analytical equipment to ISMMRRI researchers. These facilities include the Spangler Geotechnical Engineering Research Laboratory in the Civil and Construction Engineering Department, the Coal and Mineral Processing Facilities of the Chemical Engineering Department, the Coal and Mineral Microcharacterization Laboratory of the Engineering Research Institute, and the Geochemistry and Analytical Laboratories of the Geological and Atmospheric Sciences Department. The participating agricultural departments have both on- and off-campus facilities for reclamation research, including biotechnology laboratories, greenhouses, and research farms. In addition, the site of the former Iowa Coal Project Demonstration Mine has been utilized extensively for post-mining hydrology and crop research. Other research facilities available to ISMMRRI researchers include the Engineering Research Institute laboratories, the Agricultural Experiment Station, the I.S.U. Coal Preparation Plant, and many of the research facilities of the Institute for Physical Research and Technology.

Additional resources on and off campus available to the Institute include the following:

I.S.U. Coal Preparation Research Facility

The Institute for Physical Research and Technology operates a 20-70 ton per hour multi-stream experimental coal processing plant, located on the I.S.U. campus. This plant is the only research plant of its kind located on the campus of a U. S. university. The plant was dedicated in 1976 for research, including evaluation of coal beneficiation processes and techniques on a semi-commercial scale, fine coal beneficiation and sulfur reduction process development, and preparation of specially processed coals for combustion tests or as feedstocks. Experienced operators are available for collaborative research and training of students.

Coal Preparation Research Laboratories

These laboratories are equipped with reactors, autoclaves, balances, pelletizing equipment, and other items which are necessary to perform laboratory research in oxidative and caustic desulfurization, froth flotation, oil agglomeration, pelletization, briquetting, and dewatering.

Petrographic Laboratory

Equipment items include optical microscopes and furnaces for the preparation of mineral specimens by low temperature ashing. Grinding and polishing equipment necessary to the preparation of polished coal and mineral specimens is also available.

Fine Particle Characterization Laboratories

An automated image analysis system tied to scanning electron and scanning transmission electron microscopes is available for mineral association studies. Energy- and wavelength-dispersive x-ray analysis information can be gathered by a computer-controlled electron microprobe and analyzed by state-of-the-art support software. Equipment for attrition grinding, particle size characterization, BET and porosimetry measurements, and contact angle, zeta potential, and slurry viscosity measurements is available.

Chemical Analysis Laboratory

This laboratory is equipped to perform standard analyses of coal and mineral samples according to ASTM and other classical techniques.

Instrumental Analysis Facilities

The Iowa State Mining and Mineral Resources Research Institute has access to the Ames Laboratory's unique capabilities in instrumental analysis. Previous work at Ames Laboratory has resulted in the development of ultrasensitive analytical techniques, including inductively coupled plasma-atomic emission spectroscopy and various laser techniques. Other analytical techniques include x-ray fluorescence, ESCA, Auger, and spark-source mass spectrometry. Equipment for ion chromatography, gas chromatography, spectrophotometry, fluorometry, thermal analysis (by TGA, DTA, and DSC), microwave treatment, and FTIR and NMR spectrometry is readily available. Recent equipment acquisitions include an adiabatic bomb calorimeter, an iso-peribol microcalorimeter for studying adsorption reactions on surfaces of coals and minerals, and an atomic absorption/atomic emission spectrophotometer for elemental analysis.

X-ray Characterization Laboratory

This facility is equipped to characterize minerals in single crystal and powdered forms, using single-crystal diffractometers and powder diffractometers. The equipment is interfaced with computers for automated operation. In addition, an on-line x-ray diffractometer for monitoring pyrite in coal, developed with U. S. DOE funds and tested in the I.S.U. Coal Preparation Plant, is now ready for extended upgrading for other commercial applications in mineral-related industries.

Materials Analysis and Research Laboratory

The Materials Analysis and Research Laboratory (MARL) is operated by the Department of Civil and Construction Engineering and serves as a major support facility for research in the areas of science and engineering. Analytical equipment available for use includes a JEOL scanning electron microscope, a x-ray microanalyzer, a mercury porosimeter, an x-ray diffractometer, an x-ray fluorescence unit, and a thermal analyzer. Also, a patented method for determining pore size in the submicron range, called phase transition of ice porosimetry was developed by I.S.U. engineers and is available to I.S.U. researchers through MARL. The method is applicable to a wide variety of materials, including coal, soil, minerals, and geological samples.

Materials Preparation Center

The Materials Preparation Center (MPC) of the Ames Laboratory consists of a Materials Preparation Section, an Analytical Section, and the Materials Referral System and Hotline (MRSH). Materials including refractory metals, the alkaline earths, rare earth metals and compounds, and certain actinide metals can be prepared, purified, fabricated and characterized by the MPC. The Analytical Section has complete facilities for chemical and spectrographic analyses, selected services of which are available to the research community. The purpose of the Materials Referral System and Hotline (MRSH) is to accumulate information from national laboratories and commercial sources regarding the preparation and characterization of materials and to make this information available to the scientific community at no charge.

Iowa Coal Project Demonstration Mine #1

Coal mining and restoration activities have been completed at this research facility. During the course of operations 110,000 tons of coal were mined from this 40-acre site. Land that was originally pasture of marginal value has been reclaimed to topography of bench terraces suitable for intensive agricultural use. The mining method featured the use of rubber-tired scrapers to remove top soil, till, and shale overburden in a modified haul-back manner. The different overburden types were segregated during mining which enabled them to be replaced in their original order during reclamation. During terrace construction, five feet or more of non-toxic overburden, including topsoil, was replaced over the entire site. This demonstration mine site is still available for continuing agronomic and hydrologic research. Management of the site was returned to the landowner in 1988, but Iowa State University will continue to have access to this site for research purposes through a cooperative agreement with the landowner.

Childers Reclamation Research Site

As part of the mining operations, the Iowa Coal Project reclaimed 27 acres of abandoned mine lands to a field potentially valuable for hay or forage production. The area is now available as an environmental demonstration site to re-examine the effective time on reclamation projects.

Iowa State University Library

The University library collection now totals more than 1.8 million bound volumes, over 21,000 current journals and serials, more than 2 million microforms, over 115,000 maps and aerial photographs, 39,000 audio/visual items, and almost half a million photographs, as well as manuscripts and archival materials. In addition to the main collection, the library also maintains small libraries on campus in the areas of physical science, design, and economics and sociology. Its science and technology collection is regarded as one of the best in the nation.

Ames Laboratory Document Library

All research reports, theses, and publications of the scientists and engineers in the Institute for Physical Research and Technology are contained in the Document Library's reference collection.

Iowa State University Computation Center

The Iowa State University Computation Center provides research and educational computer services to the university community. The Center supplies digital computing through two separate hardware systems: a Hitachi Data Systems AS/9180 running under MVS/XA, and a VAXcluster comprised of one Digital Equipment Corporation (DEC) VAX 11/785, two DEC VAC 11/780s, and one DEC VAX 6220 running under VAX/VMS.

Rare Earth Information Center

The Rare Earth Information Center (RIC), a division of Iowa State University's Institute for Physical Research and Technology, serves the scientific and technological communities by collecting, storing, evaluating and disseminating rare earth information from various sources. RIC answers requests for information about the rare earths, publishes a free quarterly newsletter containing current and newsworthy items on rare earths, and publishes a monthly bulletin of interest to paid subscribers.

Fossil Energy Program/Ames Laboratory

The Fossil Energy Program at the Ames Laboratory, funded by the U. S. Department of Energy, focuses on projects in the areas of coal preparation, coal waste management, gas stream cleanup, and direct coal utilization. The facilities and expertise of the Fossil Energy scientists can be drawn upon to assist students and researchers interested in current and future clean coal technology.

CCE Analytical Services Laboratory

The Analytical Services Laboratory is operated by the Department of Civil and Construction Engineering (CEE). The laboratory performs all types of chemical analysis, including emission analysis, water treatment and waste water treatment analysis, and EPA toxicity testing. Instrumentation includes an atomic absorption machine, flame and graphite furnaces, gas chromatography, liquid chromatography, a wet lab, technicon auto-analyzers, and a Dohrmann DC-180 carbon analyzer (water samples only).

RESEARCH INTERACTIONS

Over the last few years, several projects dealing with coal and mineral-related research at Iowa State University have enjoyed substantial industrial and government cooperation and support. In addition, the Institute and mineral-related research groups at Iowa State University also act as a resource and perform services for industry, government, and other research centers. The summaries below are illustrative of the work and services performed in conjunction with others during fiscal year 1990.

Iowa Coal Utilization Advisory Committee

The Iowa Coal Utilization Advisory Committee was formed by the Institute to bring together representatives from both the private and public sectors who are involved in Iowa coal and mineral resources. The Committee meets on a quarterly or as-needed basis to discuss and promote coal and mineral resources. In the past year, the Committee has met to discuss research activities in coal preparation and combustion, coal and limestone contracts for the new fluidized bed combustion boilers at Iowa State University, coal demands and production levels, emission standards, and other related topics. Working relationships have been established with prominent Iowa coal- and mineral-related industries, industrial coal users, utilities, appropriate state agencies, and legislators from the General Assembly who serve on its resource-related committees.

Martin-Marietta Company

Martin-Marietta participated in the Institute's education program by giving ISMMRRI graduate students a tour of their limestone mining operation.

Iowa Coal Mining Company

The Iowa Coal Mining Company participated in the Institute's education program by giving ISMMRRI graduate students a tour of the mining operations and reclamation sites of the Star Coal Division.

Jude Coal Company

The Jude Coal Company participated in the Institute's education program by giving ISMMRRI graduate students a tour of its surface strip mine and reclamation project.

U. S. Gypsum Corporation

The U. S. Gypsum Corporation participated in the Institute's education program by giving ISMMRRI graduate students a tour of their strip mine, rehabilitation project, and wallboard manufacturing facility.

CQ Inc.

ISMMRRI was awarded a contract with CQ Inc. to conduct research on the handling characteristics of coals, in collaboration with the Electric Power Research Institute, and to perform a survey of sonic energy applications for enhancement of coal processing.

Sonic Resources, Inc.

ISMMRRI was awarded a contract with Sonic Resources, Inc., of Moline, Illinois, to improve the effectiveness of fine coal cleaning by conventional froth flotation with sonic enhancement.

Minnesota Mineral Resource Center

The Minnesota MRC asked ISMMRRI to act as a consultant to explore utilization of residual solids produced at the Metropolitan Waste Control Commission, St. Paul, Minnesota. This entails developing innovative utilization techniques for sludge ash generated by the incineration of sewage sludge. Successful completion of the project should produce an aggregate acceptable for the production of light-weight concrete.

Minnesota Power

ISMMRRI was awarded a contract with Minnesota Power to perform coal-mineral association studies and other characterizations on lignite samples. These data and samples will be forwarded to BNI Coal Ltd., a subsidiary of Minnesota Power.

Vermeer

The Vermeer Corporation is interacting with ISMMRRI-supported researchers in exploring mechanisms for developing and manufacturing a rock trencher for hard rocks.

MicroFuels

MicroFuels, an Iowa-based manufacturing company, contacted ISMMRRI to explore various avenues of industrial/university cooperation, including installation of a 5-ton/hr. centrifugal pneumatic mill (CPM) at the ISU Coal Preparation Plant to test utilization of -325 mesh coal. MicroFuels manufactures a centrifugal pneumatic mill for use in aggregate and coal grinding.

Kerr Associates

Kerr Associates, an Iowa-based company, provided ISMMRRI with a grant-in-aid for a graduate research assistant on an hourly basis to help compile a land-reclamation book in the process of being published.

Pyropower

Pyropower is the manufacturer of two circulating fluidized bed combustors (CFB) on the Iowa State University campus. Because of this unique capability and accessibility of the fluidized bed combustor units for research and testing, Pyropower will act as a subcontractor to ISMMRRI in modifying one of the Pyroflow CFB boilers for obtaining on-line isokinetic hot-gas samples for the research described below for SRI.

Southern Research Institute (SRI)

ISMMRRI is pursuing a cooperative venture among the Southern Research Institute of Birmingham, AL; the U. S. Department of Energy, Morgantown Energy Technology Center in WV; Pyropower, Incorporated, in San Diego, CA; and Iowa State University. The purpose of the one-year project is to test ceramic filter materials in one of Iowa State University's circulating fluidized bed coal-fired boilers. Filter materials of different mineral composition will be evaluated under real-world, on-line firing conditions to assess their resistance to chemical attack, thermal shock, and erosion while retaining their potential for removing particulate materials from hot gas streams. This information will help in the design of particulate filter systems for next-generation pressurized fluidized bed coal combustors.

Iowa Department of Economic Development

The Iowa Department of Economic Development provided an ISMMRRI researcher a grant to investigate bonded deposit formation in the ISU circulating fluidized bed boiler.

Associated Electric Cooperative, Inc. (AECI)

AECI, a Missouri-based cooperative provided ISMMRRI with a grant-in-aid for a graduate researcher to develop a mined-land reclamation plan of their site as a thesis project in Landscape Architecture.

SECTION V

PUBLICATIONS, PAPERS AND PRESENTATIONS

SECTION V
PUBLICATIONS, PAPERS AND PRESENTATIONS

The research activity of graduate students, faculty, and staff supported by ISMMRRI is documented by publications and papers in the field of mining and mineral resources. A list of these for Academic Year 1989-1990 is presented below.

Graduate Student Theses

Glenn Norton
"Precipitation of Jarosite-Type Double Salts from Spent Acid Solutions from a Chemical Coal Cleaning Process"
M.S. thesis
Department of Civil and Construction Engineering, Spring 1989

Thad Slaughter
"The Role Precipitating Gypsum Plays and the Accumulation of Organic Matter in Saline Solutions and the Effects of Organic Matter on the Dehydration of Gypsum"
Ph.D. thesis
Department of Geological and Atmospheric Sciences, Summer 1990

Krista McGowan
"Thermochemical Conditions for the Formation of Archean-Lode Gold Mineralization at Atlantic City-South Pass, Wyoming"
Ph.D. thesis
Department of Geological and Atmospheric Sciences, Summer 1990

ISMMRRI Publications (Academic Year 1989-1990)

Brown, R. C. and N. Christofides, "Attrition and Fragmentation of Coal-Water Mixtures in Fluidized Beds," in Processing and Utilization of High-Sulfur Coals III, R. Markuszewski and T. D. Wheelock, eds., Elsevier, Amsterdam, 1990, pp. 573-583.

Bryndzia, L. T., S. D. Scott and P. G. Spry, "Sphalerite and Hexagonal Pyrrhotite Geobarometer: Correction in Calibration and Application," Economic Geology, 85, 194-197 (1990).

Cao, X., S. M. Richardson, and C. K. Richardson, "Dissolution of MoO_2 and Complexing of Molybdenum in NaCl Solutions at Elevated Temperatures and Pressures," submitted to Geochimica et Cosmochimica Acta, May, 1990.

Dawson, M. R., D. J. Miller and W. H. Buttermore, "Bonded Deposit Formation During High-Sulfur Coal Utilization in a Circulating Fluidized Bed Boiler," in Processing and Utilization of High-Sulfur Coals III, R. Markuszewski and T. D. Wheelock, eds., Elsevier, Amsterdam, 1990, pp. 597-610.

Kramer, M. J. and K. E. Seifert, "Strain-Enhanced Diffusion in Feldspar," in Advances in Physical Geochemistry, 9, J. Ganguly, ed., Springer-Verlag, in press.

Kutz, K. B. and P. G. Spry, "The Genetic Relationship Between Upper Mississippi Valley District Lead-Zinc Mineralization and Minor Base Metal Mineralization in Iowa, Wisconsin and Illinois," Economic Geology, 84, 2139-2154 (1989).

Markuszewski, R. and T. D. Wheelock, Eds., Processing and Utilization of High-Sulfur Coals III, Coal Science and Technology 16, Elsevier, Amsterdam, 1990, 814 pp.

Norton, G.A., W. H. Buttermore, and R. Markuszewski, "Trace Elements in Coal and Coal Wastes," special report prepared for the Electric Power Research Institute, December, 1989.

Norton, G. A. and R. Markuszewski, "Trace Element Removal During Physical and Chemical Coal Cleaning," Coal Preparation, 7, 55-68, (1989).

Norton, G. A., R. G. Richardson, and R. Markuszewski, "Precipitation of Jarosite Compounds as a Method for Removing Impurities from Acidic Wastes from Chemical Coal Cleaning," submitted to Environmental Science and Technology, January, 1990.

Norton, G. A., R. G. Richardson, R. Markuszewski, and A. D. Levine, "Precipitation of Iron, Sodium, and Potassium Impurities From Synthetic Solutions Modeling Spent Acid Streams From a Chemical Coal Cleaning Process," Fuel Science and Technology International 8(7), 793-827 (1990).

Oh, J. S. and T. D. Wheelock, "Reductive Decomposition of Calcium Sulfate with Carbon Monoxide: Reaction Mechanism," Ind. Eng. Chem. Res., 29, 544-550, (1990).

Spry, P. G., "Geochemistry and Origin of Coticules (Spessartine-Quartz Rocks) Associated with Metamorphased Massive Sulfide Deposits," in Regional Metamorphism of Ore Deposits, L. T. Bryndzia and P. G. Spry, eds., VSP, in press.

Spry, P. G., C. K. Richardson, M. S. Koellner, and H. Jones, "Thermochemical Changes in the Ore Fluid During Deposition at the Denton Mine, Cave-in-Rock Fluorspar District, Illinois," Economic Geology, 85, 172-181 (1990).

Spry, P. G. and J. D. Wonder, "Manganese-Rich Garnet Rocks Associated with the Broken Hill Lead-Zinc-Silver Deposit, New South Wales, Australia, Canadian Mineralogist, 27, 275-292 (1989).

Straszheim, W. E. and R. T. Greer, "Insights on Pyritic Sulfur in Coal from Automated Image Analysis," in Processing and Utilization of High-Sulfur Coals III, R. Markuszewski and T. D. Wheelock, eds., Elsevier, Amsterdam, 1990, pp. 11-19.

Straszheim, W. E. and R. Markuszewski, "Engineering Insights from Automated Image Analysis of Mineral Matter in Coal," in Mineral Matter and Ash Deposition from Coal, R. W. Bryers and K. S. Vorres, eds., Engineering Foundation, New York, NY, 1990, pp. 53-61.

Straszheim, W. E. and R. Markuszewski, "Assessment of Coal Cleanability for Various Beneficiation Processes by SEM-Based Automated Image Analysis," Fuel Processing Technology, 24, 445-451 (1990).

Straszheim, W. E. and R. Markuszewski, "Evaluation of Physical Coal Beneficiation Using Computerized Microscopy," in Process Mineralogy IX, 1990, in press.

Mineral-Related Publications

The research activities of graduate students, faculty, and staff associated with ISMMRRI but funded by other sources are documented by additional publications and papers in the field of mining and mineral resources. A list of selected mineral-related publications for Academic Year 1988-1989 is presented below.

D. J. Akers, G. A. Norton, W. H. Buttermore, and R. Markuszewski, "Trace Elements in Coal and Coal Wastes," EPRI GS-6575, Electric Power Research Institute, Palo Alto, California, December, 1989.

G. Burnet, N. R. Fronczak, and M. J. Murtha, "Use of High-Sulfur Coal By-Products in the Lime-Sinter Process for Al_2O_3 Recovery From Fly Ash," in *Processing and Utilization of High-Sulfur Coals - III*, R. Markuszewski and T. D. Wheelock, Eds., Coal Science and Technology 16, Elsevier, Amsterdam, 1990, pp. 781-790.

W. H. Buttermore and B. J. Slomka, "The Effect of Sonic Treatment on the Floatability of Oxidized Coal," *International Journal of Mineral Processing*, submitted, 1990.

W. H. Buttermore, B. J. Slomka, and M. R. Dawson, "Sonic Enhancement of the Physical Cleaning of Coal," *Proceedings 5th Annual International Pittsburgh Coal Conference*, Pittsburgh, PA, September 12-16, 1988, pp. 431-443.

J. A. Chesley and G. Burnet, "Sulfate-Resistant Portland Cement from Lime-Soda Sinter Process Residue," *Materials Research Society*, (Symposium Proceedings, Boston, Massachusetts, November 30 - December 5, 1987), 113, 163-171 (1988).

C. D. Chriswell, N. D. Shah, and R. Markuszewski, "Countercurrent Washing of Pittsburgh No. 8 Coal After Leaching With Molten Mixtures of Sodium and Potassium Hydroxides," submitted to *Separation Science and Technology*, 1990.

C. D. Chriswell, N. D. Shah, S. M. Kaushik, and R. Markuszewski, "Chemical Cleaning of Coal by Molten Caustic Leaching After Pretreatment by Low-Temperature Devolatilization," *Fuel Processing Technology*, 22, 25-39 (1989).

C. D. Chriswell, I. Ogawa, M. J. Tschetter, and R. Markuszewski, "Effect of Hydrofluoric or Hydrochloric Acid Treatment on the Ultrasonic Extraction of Organic Materials from Fly Ash for Chromatographic Analysis," *Environmental Science & Technology*, 22(12), 1506-8 (1988).

C. D. Chriswell, N. D. Shah, S. M. Kaushik, and R. Markuszewski, "Chemical Cleaning of Coal by Molten Caustic Leaching After Pretreatment by Low-Temperature Devolatilization," *Fuel Processing Technology*, 22(1), 25-39 (1989).

C. D. Chriswell, N. D. Shah, and R. Markuszewski, "Recovery and Regeneration of Caustic for Use in Cleaning of Coal by the Molten Caustic Leaching Process," *Proceedings 5th Annual International Pittsburgh Coal Conference*, Pittsburgh, PA, September 12-16, 1988, pp. 446-460.

M. R. Dawson and C. E. Jacobson, "Geochemisty and Origin of Mafic Rocks from the Pelona, Orocopia, and Rand Schists, Southern California," Earth and Planetary Science Letters, 92, 371-385 (1989).

J. Drzymala and T. D. Wheelock, "Determining the Oil Agglomeration Characteristics of a Coal Suspension by Monitoring Turbidity Changes," in Processing and Utilization of High-Sulfur Coals - III, R. Markuszewski and T. D. Wheelock, Eds., Coal Science and Technology 16, Elsevier, Amsterdam, 1990, pp. 781-790.

J. Drzymala, R. Markuszewski, and T. D. Wheelock, "Agglomeration with Heptane of Coal and Other Materials in Aqueous Suspensions," Minerals Engineering, 1(4), 351-358 (1988).

C.-W. Fan, R. Markuszewski, and T. D. Wheelock, "Separation of Coal/Pyrite Mixtures by Agglomeration With Heptane," in Processing and Utilization of High-Sulfur Coals - III, R. Markuszewski and T. D. Wheelock, Eds., Elsevier, Amsterdam, 1990, pp. 265-278.

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