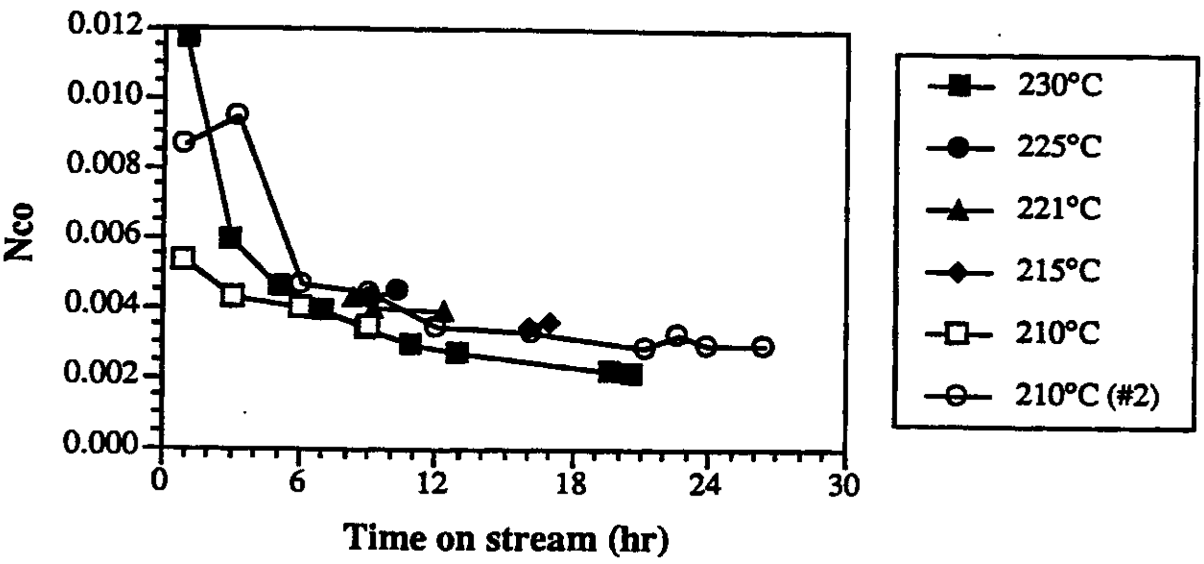

Figure 23. Activity versus time for Fe/K-A-401 at 210-230°C.

temperature tests are necessary if deactivation was to be minimized.

The sample was re-reduced in hydrogen at 400°C for about 24 hours prior to reacting further. This extended reduction time was necessary to obtain a state of catalyst in which no more (or very little) methane was being produced. Subsequent re-reductions on the potassium-promoted catalysts were done for a standard length of 20 hours. This compares to reduction times for the unpromoted catalysts of only two hours to obtain similar states.

Reaction temperatures for continued runs on Fe/K-A-401 ranged from 200 to 230°C. However, at 200°C the activity was too low to measure. Figure 23 shows the activity versus time curves at various temperatures. The two sets of data with the highest initial activities were obtained (following reduction in hydrogen) at 210 and 230°C. The time zero point is the time at which the reactor reached the given temperature. Although sample data for each temperature do not cover the full range, they do indicate that after several (about 9) hours on stream the activity at each temperature reaches a value that is within about 20% of the other temperatures. For reaction sets at 210 and 230°C where data were obtained on the freshly reduced catalyst, the higher initial activity of the higher temperature leads to a lower "steady-state" value (about 20 hours). Thus, the effect of deactivation becomes more pronounced at higher temperatures.

(Fe/K-A-402, Fe/K-A-403) Figure 24 shows the data from three different samples all reacted at 230°C at H_2/CO ratio of 3/1. The catalyst histories for the other two samples (Fe/K-A-402 and Fe/K-A-403) are given here. Hydrogen chemisorptions had been performed on the fresh Fe/K-A-402 catalyst. Reaction runs were then commenced at 210°C for 36 hours followed directly by reaction at 230°C. Fe/K-A-403 had been reacted at 210°C for 12 hours, reduced for 12 hours at 400°C and then reacted at 230°C.

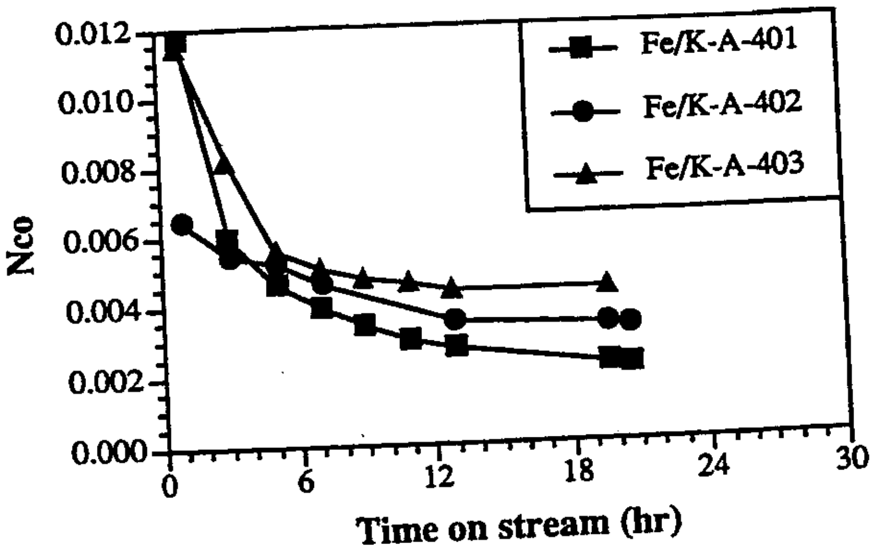

Figure 24. Specific activity versus time for Fe/K-A-401, 402, and 403 at 230°C and 3/1 H_2/CO.

As seen in Fig. 24, the two freshly reduced samples (401 and 403) have similar initial activities but 401 drops to a significantly lower value than 403. After 20 hours at 230°C, 401 activity is about half of the measured activity for 403. The fact that catalyst 403 had previously been reacted at 210°C may be the reason for the difference, even though the catalyst was reduced between the two runs. The conditions which the catalyst is subjected to initially appear to have a large impact on the "long-term" (i.e., 20 hour) state of the catalyst.

After reaction for 36 hours at 210°C, the temperature for catalyst Fe/K-A-402 was increased to 230°C. While the initial activity was only about half that for the other two catalysts, the activity after 3 hours was similar to catalyst 401 and for the rest of the time shown was intermediate between the other two catalysts.

(Fe/K-A-404) Another potassium-promoted sample (Fe/K-A-404) was used to measure activities as a function of H_2/CO ratio so that an estimate of the reaction orders for hydrogen and CO could be obtained, similar to what was done previously for the unpromoted catalysts. The catalyst was first reacted at 3/1 H_2/CO at temperatures ranging from 220-240°C so that the data for determining reaction orders would not affected by errors introduced by using different samples. Subsequent treatment at 2/1 H_2/CO (with 1 part He diluent) showed a stronger effect of deactivation on the activity of the catalyst. No further reaction experiments were performed with the potassium-promoted catalyst since kinetic data could not be obtained without significant effects of deactivation.

B. Task 2: Study of Carbon Reactivities and Types

No experiments are scheduled for this task until Spring 1991. The research assistants working on this project are presently familiarizing themselves with the literature dealing with deactivation by carbon.

C. Task 3: Mathematical Modeling of Catalyst Deactivation by Carbon

This task is scheduled to begin in the Spring 1991.

D. Technical Communication and Miscellaneous Accomplishments

During the first period, the PI hired two graduate assistants to work on this project: Mr. Mike Bayles, an MS candidate who is assigned the task of designing and constructing the computer-automated reactor system, including the writing/testing of software and Mr. Scott Eliason, a Ph.D. candidate who is assigned to carry out much of the kinetic/deactivation experiments, the study of carbon reactivities and types and a portion of the mathematical modeling. The PI also attended the 192nd Meeting of the ACS in Anaheim held September 7-12 at which he chaired a symposium dealing with CO hydrogenation; Mr. Rameswaren of the BYU catalysis lab presented a paper dealing with CO hydrogenation on carbonyl-derived Fe/alumina catalysts. Professor Jim Goodwin of the University of Pittsburgh also visited our laboratory on September 11th and presented a seminar dealing with the effects of promoters in CO hydrogenation.

During the second quarter the PI and several students attended the 2nd Annual Symposium of the Western States Catalysis Club held March 26-27 in Salt Lake City. The meeting included several papers pertinent to this study; the PI also had the opportunity to visit with Michel Boudart about progress in catalysis and combustion research at BYU and Stanford. Professor Robert Merrill of Cornell visited BYU Thursday February 5th, presented a college lecture, and conversed with the PI regarding mutual interests in catalysis and surface science research. On March 9th Professor John Sears, Chairman of Chemical Engineering at Montana State University, visited BYU to discuss with the PI research in the new Combustion Center and the BYU Catalysis Laboratory. Mike Bayles, an M.S. graduate student in Chemical Engineering, and Dr. Bartholomew published an article dealing with the computer-automated reactor system in the Macintosh Journal [46].

The BYU Catalysis Laboratory hosted Professor James Schwarz and Dr. Bob Huang of the University of Syracuse May 13-15. Professor Schwarz presented a seminar dealing with the effects of nickel catalyst preparation on chemical makeup and catalytic activity; discussions focused on catalyst preparation/characterization, TPD/TPSR measurements, and CO hydrogenation catalysis.

The PI and several graduate students, including Mr. Scott Eliason, attended the Tenth North American Meeting of the Catalysis Society held May 17-22 in San Diego; Dr. Bartholomew presented a paper dealing with CO hydrogenation on clean polycrystalline cobalt; Mr. Won Ho Lee discussed the results of a TPSR study of hydrogen with adsorbed CO on cobalt/alumina catalysts. Dr. Bartholomew also served as Program Chairman for the meeting. The technical program, which included 204 oral papers and 80 poster papers, was very stimulating. There were several papers presented in the Symposium on Catalyst Deactivation that were pertinent to this study.

The PI also attended the Am. Soc. Eng. Educ./Eng. Res. Centers Meeting June 24-25 in Reno at which he presented a paper on characterization of coals in the Advanced Combustion Engineering Research Center at BYU and the University of Utah. The following day (June 26th) Dr. Fred Steffgen, our DOE/PETC Project Manager, visited our laboratory and reviewed this DOE/PETC research program.

The Principal Investigator, Calvin H. Bartholomew, attended the International Conference on Catalyst Deactivation held September 29-October 1 in Antwerp, Belgium where he presented an invited plenary lecture on "Mechanisms of Nickel Catalyst Poisoning". He also visited the Eindhoven University of Technology on October 2 where he presented a seminar on "The Effects of Crystallite Size on the Physical, Chemical and Catalytic Properites of Iron/Carbon Catalysts," and visited with Hans Niemantsverdriet (Host), V.H.F. de Beer, A.M. van der Kraan and other scientists working in Catalysis. On October 3 he visited John Geus at the University of Utrecht and discussed mutual interests in catalysis and surface science research. On October 14-16 Professor Bartholomew attended the Sixth DOE Catalysis and Surface Chemistry Research Conference held in Gaithersburg, MD, presented a poster paper dealing with BYU's work on effects of crystallite size and support on the adsorption and catalytic properites of cobalt/alumina, and interacted with many other well-known scientists in the fields of catalysis and surface science. He also attended the 1987 Annual Meeting of the AIChE held November 15-20 in New York, chaired a session on catalyst deactivation, presented a paper on "Advanced Combustion Modeling at ACERC", and coauthored a paper on "Effects of Loading and Reduction Temperature/Time on the CO Desorption and CO Hydrogenation Kinetics on Cobalt/Titania" presented by Mr. W.H. Lee at the Symposium on Fundamentals of Catalysis. The BYU Catalysis Laboratory hosted Professor Eric Suuberg from Brown University on November 24th and discussed work on coal char characterization and oxidation, which is the subject of a research supported by a contract through the new NSF-supported Combustion Center at BYU; Professor Suuberg presented a seminar on "Active Sites and their Role in Oxidation."

A new graduate student, Pierre Smith, was hired during the fifth quarter for purposes of characterizing the iron catalysts in study by Moessbauer spectroscopy. During the past year Mr. Smith became familiar with the literature and techniques related to this spectroscopy and has begun experimentation.

During the seventh quarter the Principal Investigator and several students attended and participated in a symposium on "Catalysis in Fuel Conversion" co-sponsored by the Western States Catalysis Club and the American Chemical Society and held March 28th in Las Vegas. Mr. Byron Johnson presented a paper on the "Structural and Chemisorptive Behavior of Ultrathin Cobalt Overlayers on W(100) and W(110);" Mr. Troy Elkington presented a paper on "Ammonia Oxidation over Monolithic-Supported Platinum and Platinum-Rhodium Catalysts."

During the eighth quarter Dr. Bartholomew and Mr. Scott Eliason attended the Ninth International Catalysis Congress held June 26-July 1, 1988 in Calgary, Canada at which the PI presented a paper dealing with CO hydrogenation on Cobalt Borides and co-chaired a session on CO hydrogenation. The PI also attended the University Coal Research Contractors Meeting held July 25-28, 1988 in Pittsburgh at which he presented a talk on recent results obtained under the present contract.

The Principal Investigator (Calvin H. Bartholomew) attended and presented a paper dealing with the "Surface and Pore Properties of ANL and PETC Coals" at the 196th ACS National Meeting held September 25-30, 1988 in Los Angeles. He attended several symposia at this meeting dealing with catalysis. He was also a coauthor of a paper on "Effects of Preparation Method and Extent of Reduction on the Apparent Structure Sensitivity of Fe CO Hydrogenation Catalysts," presented at the 1988 Annual Meeting of the American Institute of Chemical Engineers held November 27-December 2, 1988 in Washington D. C. The BYU Catalysis Laboratory hosted Professor George Gavalas of the California Institute of Technology on November 23, 1988; during his visit Professor Gavalas discussed with members of the laboratory interests in catalysis and coal combustion and gave a seminar dealing with coal char combustion. The PI and students attended the Western States Catalysis Club held in conjunction with the Rocky Mountain ACS Meeting on February 24th in Denver where Mr. Scott A. Eliason made a presentation on "Fischer-Tropsch Kinetics of Unpromoted and Alumina-Promoted, Unsupported Iron Catalysts" based on work from this grant. Mr. Byron G. Johnson of the BYU Catalysis Laboratory also made a presentation on "The Structure Sensitivity of Cobalt Fischer-Tropsch Catalysts" while Paul G. Clemmer (also BYU) discussed "The Effects of Ceria Addition to Rh/Alumina Catalysts on NO reduction by CO. On March 15th, Dr. Heinz Heinemann of Lawrence, Livermore Labs, the University of California at Berkeley, visited the BYU Catalysis Laboratory and Advanced Combustion Engineering Research Center, presented a seminar dealing with catalytic gasification of coal, and discussed energy and environmental-related catalysis with members of the laboratory.The PI, Calvin H. Bartholomew, his associate William C. Hecker, and 2 students attended the Eleventh North American Meeting of the Catalysis Society held May 7-11 in Dearbom, Michigan and presented 3 papers. Mr. Byron G. Johnson presented a paper on "The Structural and Chemisorptive Properties of Ultrathin Cobalt Overlayers on W(110) and W(110), Professor William C. Hecker discussed "The Effects of Molybdena and Ceria Addition to Supported Rh Catalysts for the Reaction of Nitric Oxide with Carbon Monoxide, and Professor Calvin H. Bartholomew presented a poster paper on "Adsorption, Catalytic and Electronic Properties of Dilute Cobalt Alumina Catalysts. The PI presented a short course on Catalyst Deactivation to several students and faculty at BYU on May 24-26 and to 50 professionals at Amoco on May 31-June 2. Professors Bartholomew and Hecker and 2 students also attended an ACERC-sponsored conference held June 14th at Snowbird on Spectroscopy of Coal. Professor Bartholomew also attended the 4th Japan-China-USA Symposium on Catalysis held July 3-7, 1989 in Sapporo, Japan and presented a paper on "Temperature-Programmed Reaction of Hydrogen and Water with Preadsorbed Methane and Ethane on Nickel.

The Principal Investigator, Calvin H. Bartholomew, his associate William C. Hecker, and several students attended Advances in Catalytic Chemistry held October 1-6 at Snowbird, Utah. The PI presented an invited paper dealing with "Effects of Structure on CO Hydrogenation on Alumina-supported Cobalt and Iron."

The Principal Investigator, Calvin H. Bartholomew, attended the International Chemical Congress of the Pacific Basin Societies in Honolulu, Dec. 17-22 where he presented three papers. The PI, one associate and several students attended the Rocky Mountain Fuel Society and Western States Catalysis Club joint meeting on March 1 and 2 in Salt Lake City where they presented several papers, one of which was a paper by Mr. Scott Eliason on the "Kinetics of FT Synthesis on Unsupported Iron Catalysts," based on this contract work.

The PI and Scott Eliason attended the 1990 Spring National Meeting of the AIChE in Orlando, Florida, March 17-21 where the PI presented two invited papers at the Symposium on Applications of Fischer-Tropsch Synthesis dealing with "Hydrogen Adsorption on Supported Cobalt, Iron and Nickel" and "Recent Technological Developments in Fischer-Tropsch Catalysis". The PI also attended the 7th BES Catalysis and Surface Chemistry Research Conference held March 25-28 in Gaithersburg, Maryland where he presented a poster paper on "Effects of Dispersion and Support on Adsorption, Catalytic and Electronic Properties of Cobalt/Alumina CO Hydrogenation Catalysts." He also hosted the annual ASME Corrosion Meeting April 5-6 at BYU which focused on coal mineral transformations at which he presented a paper on CaO catalysis of char oxidation. On April 30 Professor Bartholomew presented a short course on Catalyst Deactivation at Catalytica Associates; he also presented a 3-day course on the same subject at Shell R&D in Houston on May 21-23 and visited with Shell personnel on May 24th. On May 7th the Catalysis Laboratory hosted Dr. Lynn Slaugh, distinguished scientist at Shell.

During August 6-10 Professors Bartholomew and Hecker presented a short course on Heterogeneous Catalysis and Catalyst Deactivation to 30 professionals at Corning Inc. in Corning, New York. Dr. Jeffery Ullman of Morton Chemical visited the Catalysis Laboratory on August 31st. During Fall 1990 Mr. Scott Eliason presented a paper related to the contract on "Deactivation by Carbon of Iron Fischer-Tropsch Catalysts" at the 1990 National AIChE Meeting in Chicago (Nov. 11-16) and two papers [49, 50] related to the contract were accepted for publication. The latter of these will be presented at the 5th International Symposium on Catalyst Deactivation to be held June 24-26, 1991 at Northwestern.

IV. CONCLUSIONS

1. Hydrogen adsorption at 100°C on Fe, Fe/alumina and Fe/K/alumina catalysts apparently provides the best measure of the iron metal surface area.
2. Unsupported, unpromoted iron catalysts prepared from decomposition of iron nitrate are apparently thermally stable when reduced at 300°C and run at reaction temperatures of up to 250°C in FT synthesis; however, they suffer considerable loss of surface area and heated to 400°C for extended periods and lose essentially all of their surface area when exposed to FT synthesis conditions and are then rereduced at either 300 or 400°C, probably as a result of extensive sintering during the decomposition in hydrogen of the iron carbide formed previously during synthesis.
3. Carbon forms play important roles as intermediates, catalyst additives and deactivating species in Fischer-Tropsch synthesis on iron catalysts. Deactivation may be due to poisoning or fouling of the surface by atomic carbidic carbon, graphitic carbon, inactive carbides or vermicular forms of carbon, all of which derive from carbidic carbon atoms formed during CO dissociation. While the study did not focus thus far on the chemistry of the carbon species responsible for deactivation, some important observations can be made to this end.

   a. During re-reduction of both unpromoted and K-promoted spent catalysts, methane was produced indicating that hydrogen was reacting with carbon deposited during reaction on the catalyst surface. One difference between the two catalyst types of this study (unpromoted and potassium-promoted) was the length of time required to remove the carbon. After only six hours of rereduction at 400°C the unpromoted catalyst did not produce any observable methane while the promoted catalyst produced a significant amount of methane even after 24 hours of reaction with hydrogen. Thus, it can be concluded that the surface of the potassium-containing catalyst contained more carbon after reaction, a result in agreement with previously reported work [50].

   b. Based on surface area measurements (hydrogen chemisorptions) the catalyst with 1% alumina did not sinter as a result of reaction. This was in distinct contrast to the alumina-free catalyst initially used that sintered so completely upon re-reduction in hydrogen as to lose nearly 100% of its surface area. As for the potassium-promoted catalyst, SA measurements indicate a decrease in active metal sites of around 35% but it is not known whether this is due to sintering or unreduced carbon/carbide. It is more likely due to the presence of refractory carbons that are not easily removed during rereduction.

   c. The deactivation of unpromoted 99%Fe/1% alumina catalysts during the first 30-50 hours of FT synthesis is apparently first order in catalyst activity.

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