Reprinted from:
"35th Ann. Proc. Electron Microscopy Soc. Amer." Boston, Mass., 1977. G. W. Bailey (ed.).

ELECTRON OPTICAL STUDIES OF CATALYZED GASIFICATION OF GRAPHITE

A. Szirmae, C. G. Shirley and R. M. Fisher

U.S. Steel Corp., Research Laboratory, Monroeville, Pa. 15146

The pronounced catalytic effect of small particles of iron and other elements and compounds on the reaction of carbonaceous solids with various gases (O_2, CO-CO_2, H_2O-H_2) has been documented by a number of investigators reporting on thermogravimetric studies on bulk samples (1,2) and on optical and electron microscope observations of pitting or surface channeling by rapid local attack adjacent to sub-micron iron particles (3,4). In this work, emphasis has been placed on detailed electron microscope studies of Fe particle channeling on graphite exposed to "wet" and "dry" hydrogen (P_{H_2O} 3×10^{-2} $\sim6\times10^{-6}$) at temperatures from 650°C to 1200°C.

Flakes of freshly cleaved Ticonderoga graphite are coated with 40 - 1000 Å of Fe by vacuum deposition which forms spherical particles during subsequent annealing at 750°C. Specimens are reacted for from 5-90 minutes in H_2-H_2O-He atmospheres and examined in the USS-MVEM or an ETEC-SEM. SEM and MVEM observations of the surface channels resulting from the Fe-catalyzed reaction producing CO and/or CH_4 have revealed variations in their nature which are dependent upon temperature and P_{H_2O} level. Very narrow, parallel-sided channels, initiated at ledge steps on the cleaved graphite, take on <11$\bar{2}$0> directions at lower temperatures, whereas at higher temperatures broad wavy tracks are left by iron particles which continue to grow by the addition of other iron particles encountered in their path.

At 1100°C in very dry hydrogen, the edges of the very broad reaction zones are wetted by the iron phase forming thin single crystal ribbons. The electron diffraction patterns of these particles have not been identified. Spectrochemical analysis in the SEM of specimens reacted above 900°C has revealed the presence of a large amount of Si in the particles which may originate from impurities in the graphite or possibly from the quartz furnace tube. Measurements of the length of the longest channels on selected specimens show a variation of not more than a factor of 20 over the full temperature range in keeping with a low (∿10 kcal) activation energy.

This research is supported by ERDA, Division of Physical Research, Contract No. E(11-1)-2933.

1. H.H. Lowry, ed., Chemistry of Coal Utilizztion, Suppl. Vol., (John Wiley and Sons, Inc., New York, 1963).
2. E.T. Turkdogan and J.V. Vinters, Carbon 10, 97 (1972).
3. R.T.K. Baker and P.S. Harris, Carbon 11, 25 (1973).
4. D.W. McKee, Carbon 12, 453 (1974).

Reprinted from:
"35th Ann. Proc. Electron Microscopy Soc. Amer." Boston, Mass., 1977. G. W. Bailey (ed.).

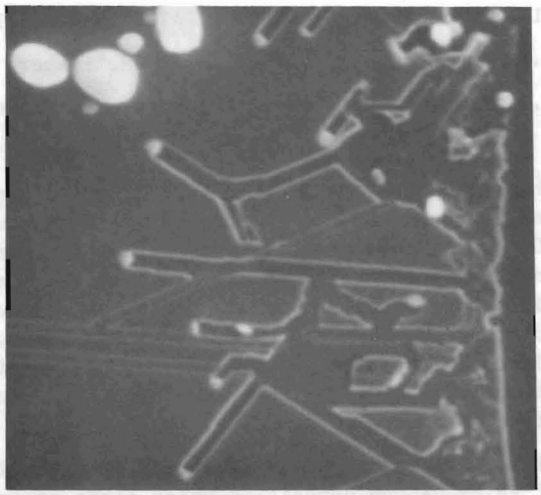

Fig. 1 - Fe-Catalyzed Reaction Zones.

(A) SEM - 14,000X;
850°C - $P_{H_2O} = 2 \times 10^{-2}$

(B) MVEM - 5,000X;
1100°C - $P_{H_2O} = 4 \times 10^{-4}$

(C) MVEM - 22,500X
1100°C - $P_{H_2O} = 10^{-5}$