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Production of Oxygen From Lunar Ilmenite

Y. Zhao and F. Shadman
Department of Chemical Engineering
The University of Arizona

Oxygen is a consumable material that needs to be produced continuously in most space missions. Its use for propulsion, as well as life support, makes oxygen one of the largest volume chemicals to be produced in space. Production of oxygen from lunar materials is of particular interest and is a very attractive possibility.

Among the proposed processes, the hydrogen and carbothermal reductions of ilmenite appear very promising. Hydrogen reduction has a relatively simple process configuration; the individual steps are relatively well studied. However, the major problem is the large heating and cooling loads required to condense the water and then heat the hydrogen to its reaction temperature. Handling and storage of large amounts of hydrogen are also a problem. Hydrogen reduction of lunar ilmenite has been studied by several investigators. Carbothermal reduction of terrestrial ilmenite has also been extensively studied, but its application to lunar ilmenite is still an open area for investigation.

The overall objectives of this study can be described as follows:

- Study of the mechanism and kinetics of carbothermal reduction of simulated lunar ilmenite using carbon and, particularly, CO as reducing agents; determination of the rate-limiting steps; investigation of the effect of impurities, particularly magnesium; search for catalysts suitable for enhancement of the rate-limiting step.
- Comparison of the kinetics of carbothermal reduction with those of hydrogen reduction; study the combined use of CO and hydrogen as products of gasification of carbonaceous solids.
- Development of new reduction methods that are based on the use of waste carbonaceous compounds for the process; development of a novel carbothermal reaction path that utilizes gasification of carbonaceous solids to reducing gaseous species (hydrocarbons and carbon monoxide) to facilitate the reduction reaction kinetics and make the process more flexible in using various forms of carbonaceous feeds.
- Development of advanced gas separation techniques, including the use of high-temperature ceramic membranes.
- Development of an optimum process flow sheet for carbothermal reduction, and comparison of this process with the hydrogen reduction scheme, as well as a general comparison with other leading oxygen production schemes; use of new and advanced material processing and separation techniques.

Experimental Method

Two reactor systems have been designed, fabricated, and put into operation: A small fluidized bed reactor for quick response times and a thermo-gravimetric reactor system with an electronic microbalance for the continuous measurement of the rate and the sample conversion. A unique method is developed for preparation of two types of ilmenite samples used in the experiments. The first type was pure synthetic ilmenite with impurity less than 0.1%. The second was synthetic ilmenite in which known and controllable amounts of impurities are introduced to simulate actual impurities found in lunar samples. A series of tests, including X-ray diffraction experiments and Mossbauer spectroscopy, were performed on synthetic ilmenite to ensure that the iron in these samples was Fe^{++}.

In each experiment, a known amount of ilmenite was exposed to the reducing gas, and the rate of conversion was monitored using both gravimetric response and continuous monitoring of the gases produced. The starting samples were fully characterized by particle size, porosity, surface area, and composition. The important reaction parameters are temperature, reducing gas composition, and flow rate.

Results and Conclusions

A typical temporal profile of conversion is shown in Figure 1.3. The results indicate the presence of three stages:

- Induction stage during which reaction is very slow. Detailed microprobe analysis of samples quenched during this stage indicate that the formation of iron nuclei is the rate-controlling step. The presence of small amounts of impurities can affect this stage.
- Acceleration stage during which rate increases progressively. The reaction rate during this stage is influenced by diffusion of reducing gas into the particles and diffusion of iron away from the reaction front.
- Deceleration stage which is primarily due to depletion of ilmenite.

The effect of temperature on the rate and the activation energies at different levels of conversion are shown in Figure 1.4. The effect of CO concentration on the rate is shown in Figure 1.5. Partially converted samples were probed using high-resolution energy dispersive X-ray analysis; the micrographs and concentration maps show the importance of iron nuclei formation and growth in controlling the rate. Sample results are shown in Figures 1.6 and 1.7.

An important finding during this phase of the project has been the determination of the rate-limiting step and the mechanism of impurities action. These results are important in developing reaction conditions and selecting catalysts to enhance the rate and lower the reaction temperature. The experimental work on the effect of magnesium, an important impurity, on kinetics is currently in progress. The reactor system is being prepared for experimental runs with hydrogen and mixtures of hydrogen and carbon monoxide as reducing gases.

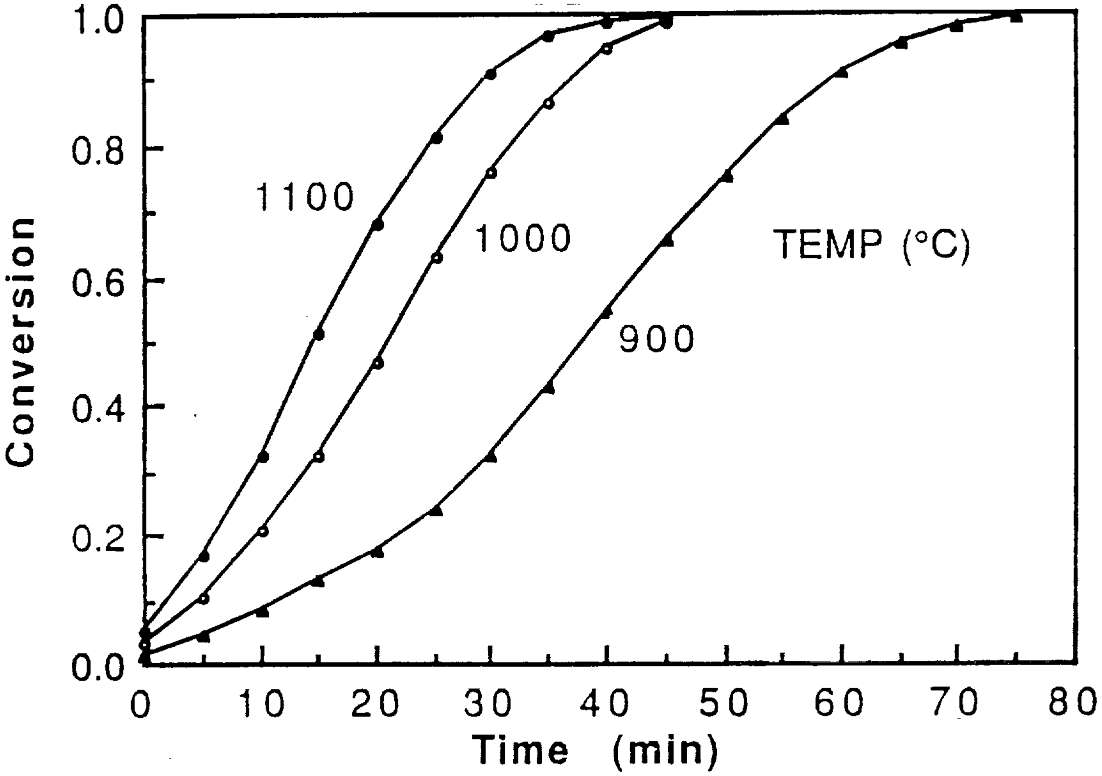

Figure 1.3 Temporal profile of synthetic ilmenite reduction by carbon monoxide.

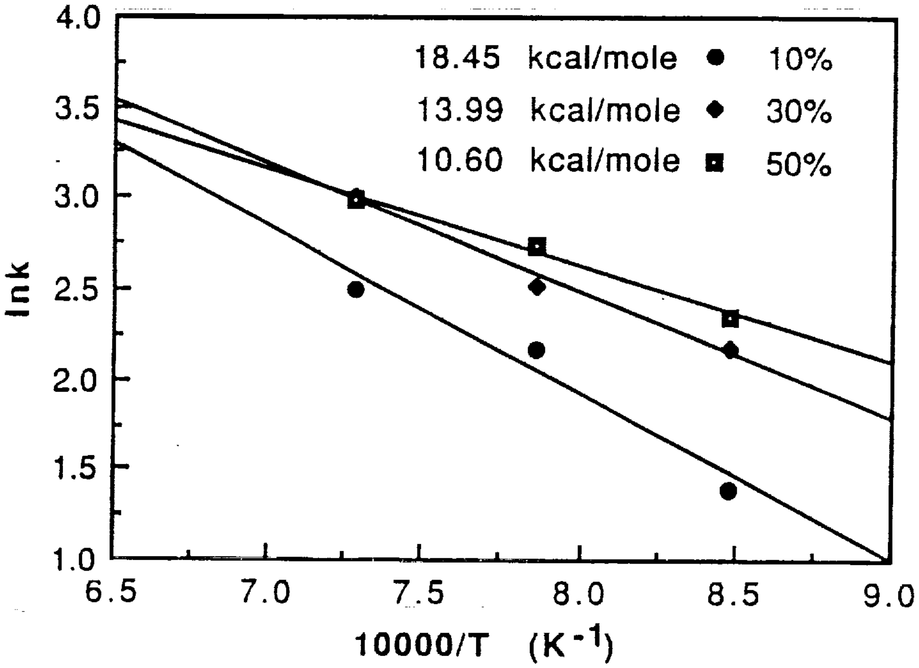

Figure 1.4 Apparent activation energy at 10, 30, and 50% conversions.

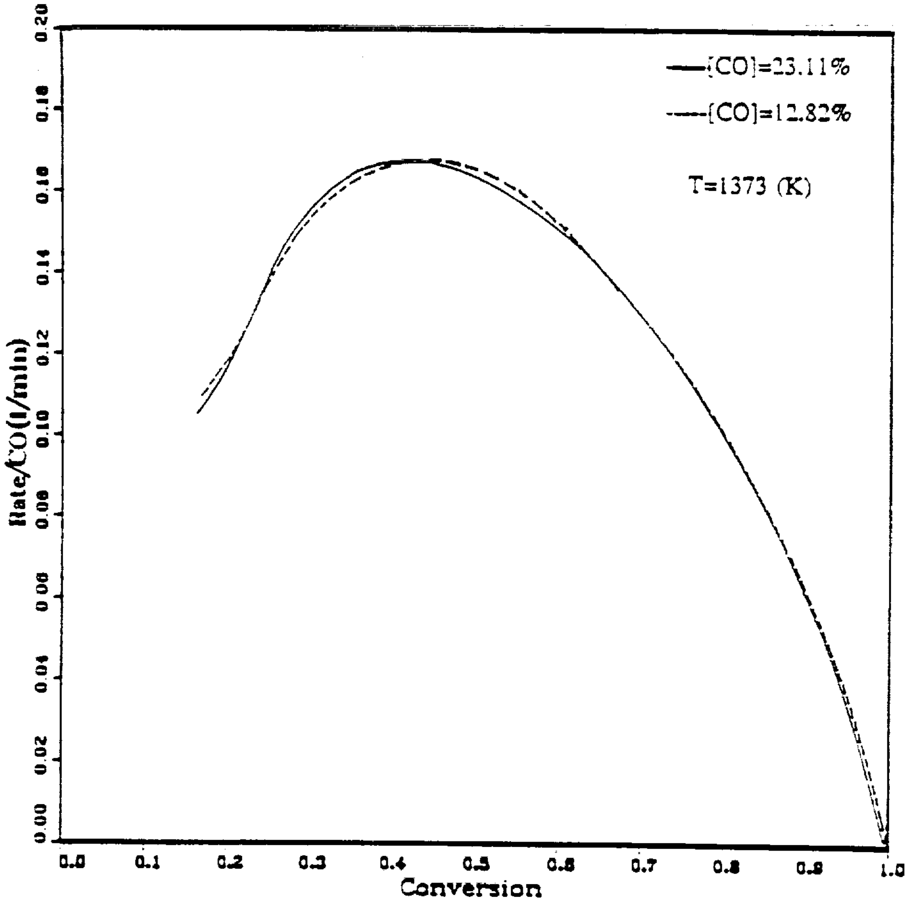

Figure 1.5 Effect of CO concentration on the rate of reduction.

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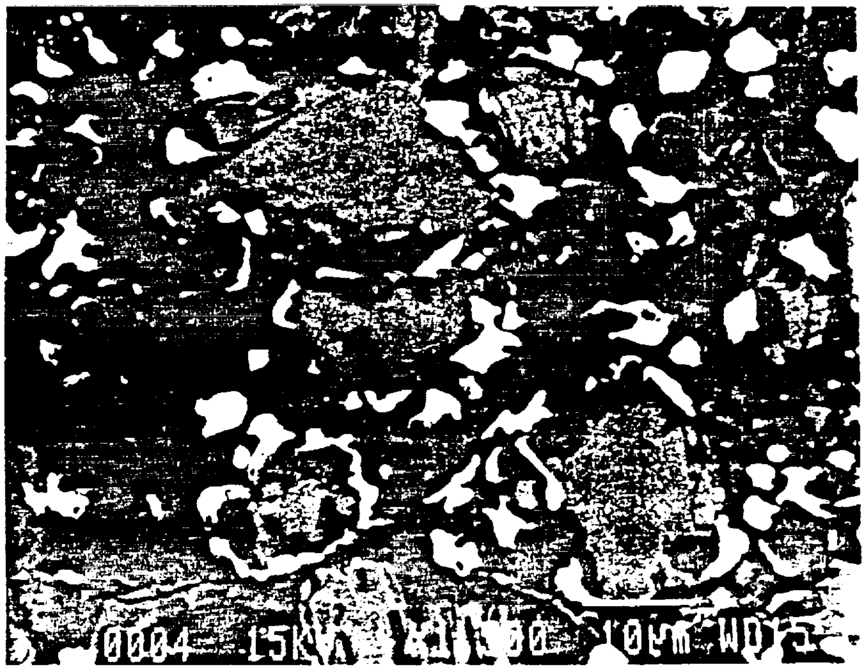

Figure 1.6 SEM backscattered electron micrograph of the polished cross section of a synthetic ilmenite flake after partial reduction at 1000° C.

Figure 1.7 SEM secondary electron micrograph of two grains of the sample shown in Figure 1.6.