In-situ observation of the cross-reaction of the oxides promoted by electron irradiation

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1. Introduction

The emission amount of carbon dioxide in the iron and steel industries because of the use of coal as reductant, heat source, carburization and so on. There are many research projects for reduction of carbon dioxide. But most of them are still depend on the carbon as reductant because blast furnace is very economical way for ironmaking. Then the limitation of the reduction of carbon dioxide are still remained.

Recently we found that several kinds of ceramics which could reduce iron-oxide with in-situ electron microscopy. They do not include carbon and no carbon dioxide produce by the reaction with iron-oxides. The analysis of the reaction with silicon was also carried out to compare the reaction process with the use of silicon base ceramics. The aim of this study is to investigate some special reductant which does not produce CO_2 by reduction of iron-oxides.

2. Experimental Procedure

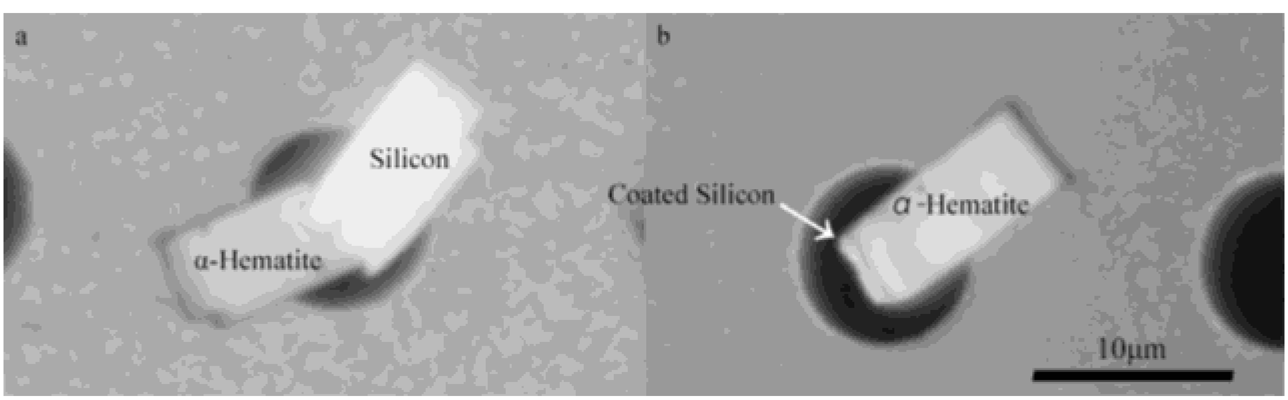
Reagent of Φ#177;-hematite and other silicon based ceramics were utilized in our study. TEM specimens were prepared using FIB. The two types of specimen was prepared, namely, the stack of two specimens to ensure contact of both materials and one material coated on hematite to analyse the reaction of interface between hematite and coated materials. The wider contact area can be get in this type of specimen but the direction of observation is limited for cross sectional analysis of the interface. Figure 1 presents optical micrographs of the specimen placed on the tip of the TEM holder.

3. Results

Figure. 2 shows the continuous observation of the precipitate at the contact point of Φ#177;-hematite and elemental silicon maintained at 973K in a stacked specimen. These photographs were captured from video images, and

several precipitates appeared in b) and then suddenly expanded after a certain period of time. The precipitates of post expansion was showed in c). The expansion was happened in the precipitates which maintained on the elemental silicon and it seemed rupture. There is the possibility that the first precipitate was reduction of hematite and next expansion was reaction with silicon. No more reaction occurred after expansion.

Fig.1 Optical micrograph of examples the specimens put on the heating stage. a) is the silica and α-hematite stacked specimen and b) is the silicon coated α-hematite to ensure the interface with silicon.



The annealing experiments outside TEM which means the condition of without assist of electron beam was also carried out. Fig.3 shows the results of the 4h annealing at 1373K in the air. The silica coated Φ#177;-hematite was used in this experiment. Small Fe precipitates was found in silica layer. This results indicated that the Fe precipitates without electron irradiation but the diffusion rate into the silica was very slow. If the large scale of bulk experiments would done, it would took long time to find the precipitation of Fe and it was found that electron irradiation was very effective.

Fig. 2 The continuous observation of the precipitate at the contact point of α-hematite and elemental silicon maintained at 973K in a stacked specimen. a) is just before the precipitation start, b) is 9min passed after a), c) is 27min passed after b) almost all precipitates expanded.

Fig. 3 The cross sectional TEM micrograph of silica coated α-hematite which annealed at 1373K for 4h in the air and b) was about 200 times enlarged Fe cluster pointed by arrow in a).