Extended Abstract of PSA-19

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In-situ observation of the interaction silicon and hematite

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(Received: June 06, 2019; Accepted: August 03, 2019)

The reduction process of the Fe_2O_3 (α-hematite) by Si was investigated with in-situ transmission electron microscopy (TEM). Hematite and other iron oxides are main component of iron ore and Si is main gangue elements of iron ore. Si reduced hematite and Fe precipitated around 700℃. The electrons due to TEM(=200keV) was not necessary condition of Fe precipitation and grew until run out of Si. The residual Si reacted with precipitated Fe after a certain period of time of electron irradiation. The reaction completed very short time less than one frame of video. One of the reacted material was identified as α-$FeSi_2$. These results indicated that the possibility of new ironmaking without CO_2 emission and silicide formation.

1. Introduction

In-situ transmission electron microscopy (TEM) is one of the powerful tool to get real time information about the change of the mechanical properties, electrical properties, radiation effects and so on. Recently we have been studied the interaction among the iron-oxides and silicon base materials with in-situ TEM using heating stage. It was found the several kinds of Si base materials reduced Fe-oxides. On the other hand, the huge amount of CO_2 emission from the iron and steel industries is one of the serious environmental problem. The materials in our study includes no carbon and no CO_2 will emit by Fe-precipitation. Our experimental results indicate that there is the possibility of CO_2 free iron making. The formation process of iron-silicide was also observed. This paper is described about the results of in-situ TEM of the reaction of the iron-oxide and Si.

2. Experimental Procedure

Reagent of α-hematite(Fe_2O_3) and Si 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 another type is Si coated hematite to analyze the reaction of interface between hematite and Si. The wider contact area can be obtained in stacked specimen but the direction of observation is limited for cross sectional analysis of the interface. Figure 1 presents optical micrographs of the

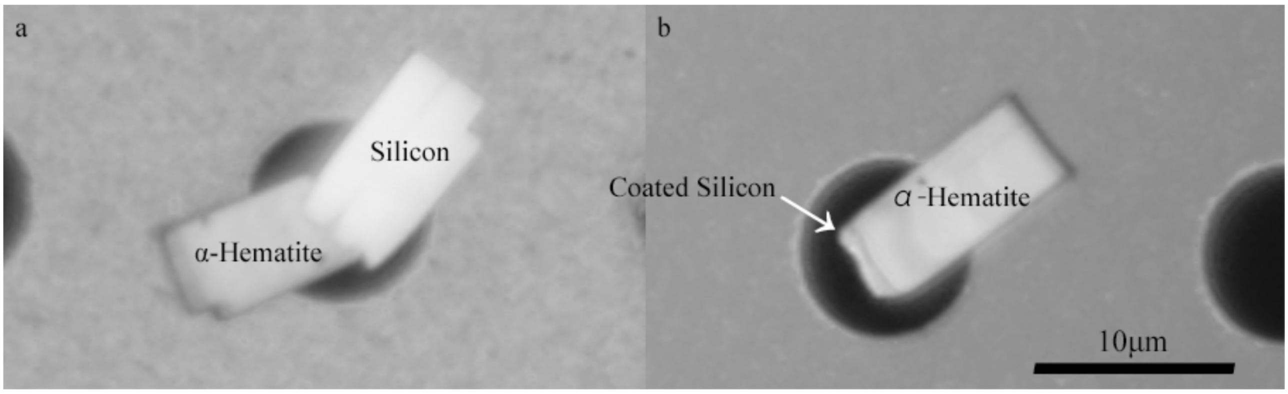

Fig.1 Optical micrograph of examples the specimens put on the heating stage. a) is the silicon and α-hematite stacked specimen and b) is the silicon coated α-hematite to ensure the interface with Si.

specimen placed on the tip of the TEM holder of Fusion series made by Protochips company. TEM of JEM-2100F was used for our study.

3. Results

The standard generation Gibbs energy of iron and silicon indicates that silicon reduce iron oxides. Figure 2 is the stacked specimen kept at 700℃ up to 30h. The precipitates were identified as Fe by EDS. The growth of the precipitates were video-recorded and condition of electron-block was also carried out. The Fe-precipitation was also caused without the assist of electron. The precipitates became larger with the decreasing the volume of silicon and grew until silicon run out. The nucleation was happened at the surface of the hematite. Very small amount of Si was detected in precipitated Fe. Disappeared silicon is considered to become silicon mono-oxide.

Most of precipitates which remained on silicon were transformed to another phase. These transformation were completed for a very short time and the assist of electron irradiation was necessary. In case of figure 2, transformation was not caused because observation was carried out with minimum dose of electron for video recording. The volume of the Si around the transformed precipitates reduced rapidly. Figure 3 shows the continuous observation of the transformation and it was completed within 1 frame of the video. The transformed particles spread on Si and absorbed neighboring particles. The transformed particle was identified as α-$FeSi_2$ by the diffraction. α-$FeSi_2$ is stable at high temperature and β- $FeSi_2$ is payed more attract attention with various physical properties as power semiconductor, light emitting device, Peltier device and so on. Finding the way of change to β phase or the condition of direct transformation to β-$FeSi_2$ is future subject.

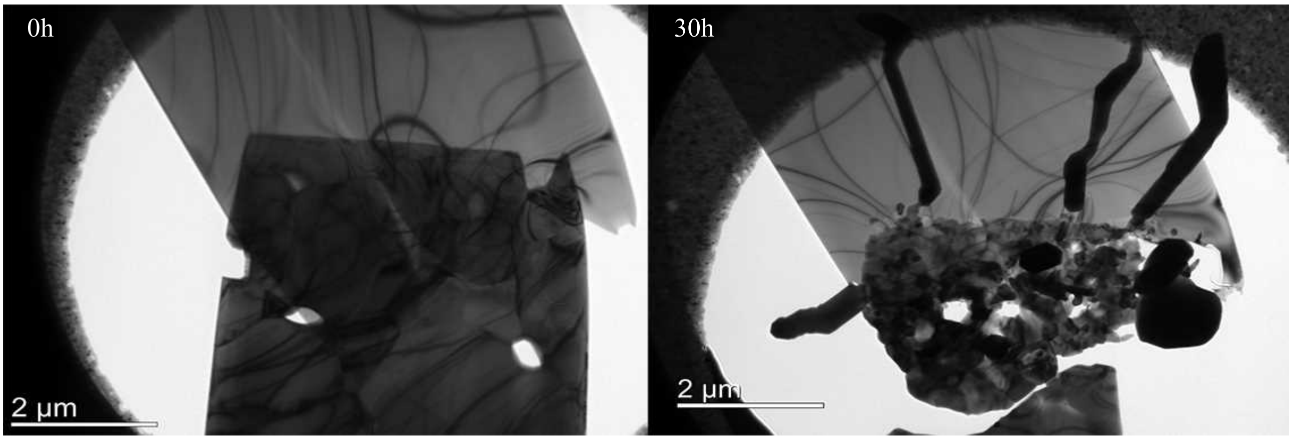

Fig.2 The growth of Fe-precipitates followed until 30h passed at 700℃.

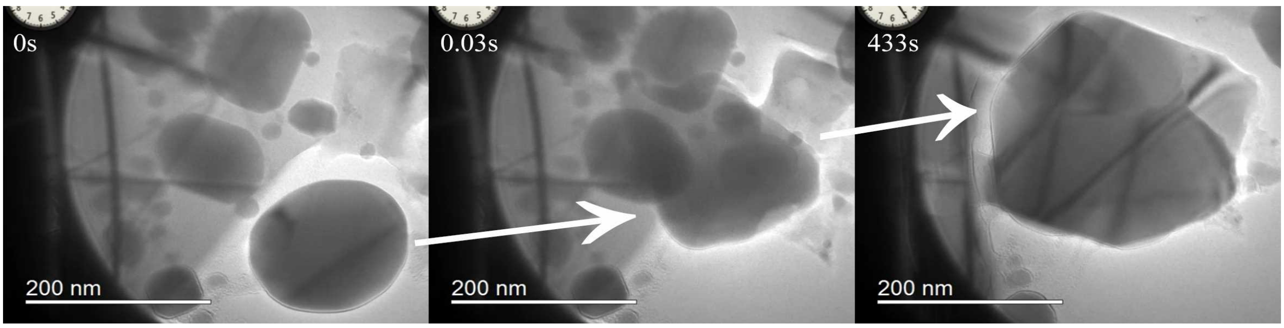

Fig.3 Silicide formed from precipitated Fe on Si with electron irradiation kept at 700℃. The transformation was pointed by arrows. Transformed precipitate was identified as α-$FeSi_2$ transformation the transformation was completed within 1 frame of the video(=0.03s) and spread with absorbing neighboring precipitates after several minutes of transformation.