A STUDY ON THE FABRICATION OF IRON POWDER FROM FORGING SCALE USING HYDROGEN

This study was conducted to investigate the effect of hydrogen content, temperature, reaction time for the reduction of forging scale which is mainly composed of hematite (Fe₂O₃). All reductive reactions were performed over the temperature range of 700 to 1200°C as well as 0.1 to 1 atm of hydrogen partial pressures. The results showed that the mechanism for the reduction of iron oxides using hydrogen gas was not a simple process, but proceeded in multiple reduction stages thermodynamically. The iron oxide was almost completely reduced to metallic iron powder with 91 wt.% of iron content in the forging scale at 0.1 atm of hydrogen partial pressure. The content of iron was however found to be increased with increasing hydrogen partial pressure from 0.1 to 1 atm with regardless of temperatures. The metallic iron powder was obtained with the mean size of 100 μm and more porous structure was observed.

Keywords: forging scale, hydrogen partial pressure, hematite, iron powder

1. Introduction

Forging is the process that deforms metal into a predetermined shape using localized compressive forces. As the metal is shaped during operation, deformation is always accomplished using hot, cold, or even warm forging processes. Generally, hot forging process has been recommend for the fabrication of industry components with a high formability ratio, low stress, low work hardening, and increased ductility. On the other hand, hot forging process will also rise to a large amount of loss of the main component due to spalling off of oxide scale when oxidation reaction takes place between the surrounding atmosphere and the metal. The oxide scale taken away from a main body during heat treatment is called as a forging scale. Unfortunately, the forging scale has not been attempted yet to be recycled and reused by many researchers and manufacturer. Therefore, this study was conducted to produce iron powder from forging scale using hydrogen gas.

The reduction of iron ore by hydrogen was widely studied and most of the reaction features were very similar to those of the reduction by carbon monoxide and many mechanisms are common to both of them [1-5]. In this study, a forging scale of industrial waste was employed as a raw material to fabricate metallic iron powder using hydrogen gas. The effect of temperature, reaction time, and hydrogen partial pressure was widely studied to determine the kinetics of iron oxide reduction by hydrogen. The characteristics of final species were examined by scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDX), and X-ray diffraction (XRD).

2. Materials and methods

Forging scales were obtained from a manufacturer of forged metal products in Korea and their chemical composition and phase were examined by X-ray diffraction (XRD) and Energy dispersive X-ray spectroscopy (EDX) shown in Fig. 1. It is noted that the forging scale is composed of three different types of iron oxides, and main peak from XRD result are shown to be hematite (Fe₂O₃). In addition, EDX analysis indicated that iron shows a large portion accounting for about 68%, and 28% for oxygen of the forging scale. The small amounts of impurities were also observed such as silicon, manganese, chromium, aluminum, etc which takes up about 4% out of total amount. Therefore, reduction reaction of iron oxides by hydrogen should be taken into account for three different oxides and other impurities of the forging scale.

A schematic diagram of experimental apparatus for the reduction of iron oxides using a gas mixture of hydrogen and argon gas is shown in Fig. 2. The powder of forging scale was put on the alumina tray that was placed on the center of an alumina reactor. Copper turning furnace was installed in front of ultra high purity (UHP) argon gas to eliminate humidity from the gas. Two flask bottles are located at the end of re-
actor to safely handle wasted gas and finally extra hydrogen gas completely was burned out. The sample tray with forging scale particles was placed in the reactor and UHP argon gas was kept to flow into the reactor until desired temperature was reached. The mixture of hydrogen and argon gas was introduced into the reactor, and then the reactor was cooled down to the room temperature under UHP argon atmosphere at each reaction time.

The overall process to fabricate metallic iron powder from the forging scale is illustrated in Fig. 3. The forging scale was spontaneously taken apart from forged metal products during operation. The scale particles collected were ball-milled for 3 hours with tungsten carbide balls leading to fine size of particles. Consequently, sieving process was conducted to screen rough particles and the mean particle size was found to be around 100 µm which is suitable in powder metallurgical process. The oxide particles were reduced by hydrogen gas on the effect of temperature, reaction time, and hydrogen content. The final species when reduction was completed were examined by XRD, EDX, and SEM to determine whether reduction reaction was successful or not.

3. Results and discussion

The reduction of iron oxides by hydrogen is known as a gas-solid reaction which occurs in various stages depending on the temperature and oxygen content shown in Fig. 4. For the temperature higher than 570°C, hematite (Fe₂O₃) is first transformed into magnetite (Fe₃O₄), then into wüstite (Fe₁₋ₓO), and finally into metallic iron (Fe). On the other hand, at temperature below 570°C, magnetite is directly transformed into iron, since wüstite is not thermodynamically stable [6].

Multiple step processes for the reduction of iron oxides by hydrogen were proposed and their data of Gibbs free energy (G) were calculated by thermodynamic program of HSC Chemistry 5.1. As stated below, hematite can be directly reduced to metallic iron, and only water vapor (H₂O) evolved in one-step process. In case of two-step process, hematite can be transformed into magnetite and metallic iron in order, and in three-step process hematite can be reduced to wüstite that is decomposed into magnetite and iron. Consequently, magnetite can be reduced to iron, and water vapor was generated at the same time. In a multiple-step process, hematite can be transformed into magnetite and it is again reduced to wüstite. The wüstite can be decomposed to magnetite once again that means the phase transformation of magnetite into wüstite will be possible to take place in a cycle leading to finally them become metallic iron.

- [U+25FC] One-step process [Fe₂O₃ →Fe]
  Fe₂O₃ + 3H₂(g) → 2Fe + 3H₂O(g)  ΔG₆₀₀°C = -1.96kcal

- [U+25FC] Two-step process [Fe₂O₃ →Fe₃O₄ →Fe]
  Fe₂O₃ + H₂(g) → 2Fe₃O₄ + H₂O(g)  ΔG₇₀₀°C = - 23.30kcal
  2Fe₃O₄ + H₂(g) → 3Fe + 4H₂O(g)  ΔG₁₁₀₀°C = -1.54kcal
Fe₂O₃ + H₂(g) → 2FeO + H₂O(g) \[ \Delta G_{700°C} = -5.62 \text{kcal} \]
4FeO → Fe₃O₄ + Fe \[ \Delta G_{600°C} = -0.21 \text{kcal} \]
2Fe₃O₄ + H₂(g) → 3Fe + 4H₂O(g) \[ \Delta G_{1100°C} = -1.54 \text{kcal} \]

Fe₂O₃ → Fe₃O₄ ↔ FeO → Fe

Fe₂O₃ + H₂(g) → 2FeO + H₂O(g) \[ \Delta G_{700°C} = -23.30 \text{kcal} \]
2FeO + H₂(g) → 3Fe + 4H₂O(g) \[ \Delta G_{800°C} = -0.16 \text{kcal} \]
4FeO → 2Fe₃O₄ + Fe \[ \Delta G_{600°C} = -0.12 \text{kcal} \]

4. Conclusions

The reduction of iron oxides by hydrogen was conducted on the effect of temperature, reaction time, and hydrogen partial pressure, and the multiple-step of reaction proposed above was thermodynamically considered. The reduction of iron oxides by hydrogen was found to be so fast less than 10 minutes using a pure hydrogen gas, and the study of reaction kinetics was therefore recommended to be measured less than 10 minutes in the further study. Moreover, iron content in the forging scale was shown to be increased with increasing hydrogen partial pressure at each temperature. Finally, the metallic iron powder reduced was observed to have an average size of 100 µm and has a porous structure due to reduction of oxygen by hydrogen.

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REFERENCES