

# Characterization of Iron Powder Produced by Reduction of Hot-Rolled Mill Scale In Hydrogen Gas

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Rolled mill scale is one of the most valuable industrial waste. The produced mill-scale briquettes are reduced by hydrogen at varying temperatures. The reduction is carried out at 800°C, 900°C, and 1000 °C and lasts for 120 minutes. The reduced samples are studied by X-ray diffraction and scanning electron microscopy. The reduction of mill scale allows the new use and development of this material to obtain sponge iron that can be reused to produce iron-based powder metallurgy components. From the SEM, EDS, and XRD analysis, it is clear that as the reduction temperature increases from 800°C to 1000°C, the amount of produced iron powder also increases at constant holding time, where the amount of iron powder produced at 800°C is 94.55%, but it is 98.22% at the reduction temperature of 1000°C. The proposed reduction process allows obtaining a sponge iron powder of an irregular morphology with different crystallite sizes as measured by the Scherrer equation, where the crystallite size increased with the reduction temperature. The minimum crystallite size value is 44.45nm at the reduction temperature of 800°C, but it equals 60.1nm at 1000°C.

**Keywords:** *Rolled mill scale, Reduction process, Crystallite size, Hydrogen atmosphere, Iron powder.*

## 1. Introduction

Due to environmental regulations, the waste management generated by hot metal and steel has become an important issue. Furthermore, with a huge demand for steel, steelmakers produced considerable amounts of mill-scale which is considered as waste materials. Consequently, an extensive study should be conducted to reuse such waste material to fulfill the needs in manufacturing aspects<sup>1</sup>. The metallurgical industry is one of the most considerable materials forming industries. In many iron and steel forming processes, solid wastes of mill-scale are generated, representing about 2% of the produced steel<sup>2</sup>. During the hot rolling process of steel, iron oxides form on the surface of the metal as scales. The scale is accumulated as waste material in all steel companies. Mill scale is often called scale, and it is the flaky surface consisting of the different types of iron oxides; iron oxide (FeO), hematite (Fe<sub>2</sub>O<sub>3</sub>), and magnetite (Fe<sub>3</sub>O<sub>4</sub>)<sup>3</sup>. It can be considered as a valuable metallurgical raw material for iron and steel-making industry<sup>3</sup>. Mill scale is one of the most valuable industrial waste due to its richness in iron<sup>4</sup>. Mill scale is a by-product from the rolling mill in steel hot rolling processes. It contains both iron in elemental form and three types of iron oxides: wustite (FeO), haematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>), and magnetite (Fe<sub>3</sub>O<sub>4</sub>). The chemical composition of the mill scale varies according to the type of steel produced and the process used. The iron content is normally 70%, with traces of non-ferrous metals and alkaline compounds, and is contaminated with lubricants, oils, and greases from

the equipment associated with rolling operations. In the whole world, 13.5 million tons of mill scales are generated annually<sup>5</sup>. Mill scale is suitable for direct recycling to the blast furnace via a sintering plant<sup>6</sup>. Approximately 90% of the mill-scale is directly recycled for steel making industry, and small amounts are used as ferroalloys in cement plants and the petrochemicals industry<sup>7,8</sup>.

The reduction of rolling mill scale to sponge iron powder is potentially a new way to take advantage of a cheap by-product of the steel-making industry, yielding sponge iron that can be reused to the electric furnace as a metallic charge for steel-making to obtain a product with a lower residual content and improved properties<sup>8,9</sup>. Other possible uses include manufacturing structural components from sponge iron powder, such as magnets and components for electric motors. High purity sponge iron powder is currently manufactured from high purity iron ore following the basic steps of magnetic separation and milling, primary reduction process, hydrogen annealing, and final milling and classification. Reducing iron oxides via gaseous and solid reductants has already been extensively studied. Benchiheub et al.<sup>10</sup> found that the best reduction of mill scale was obtained at 1050°C at 180 min in pure carbon monoxide gas. Bagatini et al.<sup>11</sup> studied the kinetics of scale reduction by carbon monoxide in the temperature ranging in 800°C-1200°C, and the results indicated that un-reacted shrinking core model with one interface under chemical reaction control fits well with the experimental data at the initial stage of reduction and the activation energy about 80 kJ/mol. Kawasaki et al.<sup>12</sup> found

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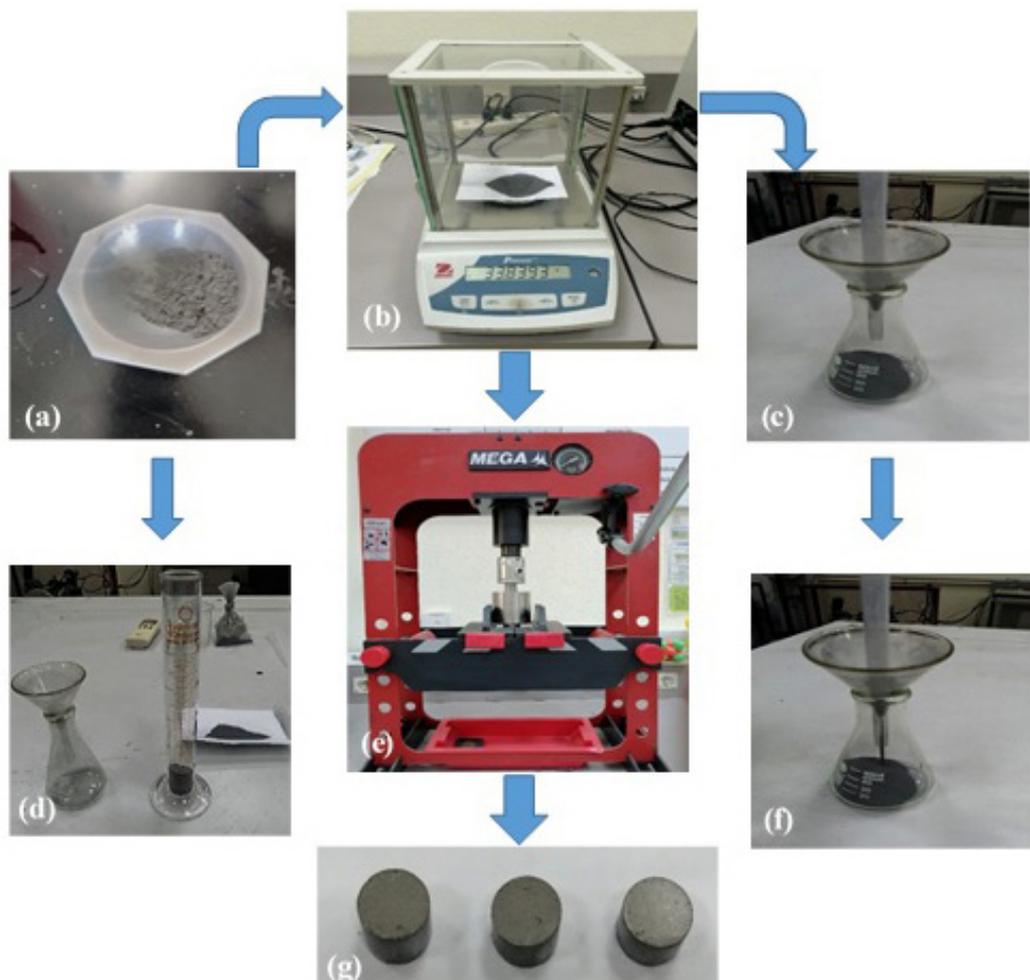
that varying the total pressure of the reduction system by hydrogen and carbon monoxide did not affect the reduction rate. Gaballah et al.<sup>4</sup> found that the degree of reduction of mill scale briquettes at constant temperature increased as the hydrogen flow rate increased. The main objective of the present study is to characterize the iron powder produced by the reduction of hot-rolled mill scale in the hydrogen gas atmosphere. SEM, EDS, and XRD analysis were used to characterize mill-scale and produced iron powder. The effect of reduction temperature on the efficiency of the reduction process and crystallite size was also investigated.

## 2. Experimental Work

### 2.1. Material and procedure

The manufacturing of steel products into blooms, sheets, billets, slabs, or bars requires working or forming in a hot strip-rolling process. During the working or forming, these products undergo cooling and washing with a high-pressure water spray that will lead to oxidation. The hot steel surface is

exposed to oxygen from the air and wash water. As a result of the exposure, thin layers of iron oxides formed on the surface are flaked off. As the steel products are rolled into the desired shape, one layer of scale is broken off and is replaced by a new scale layer. A new scale layer is generated every time the steel product is shaped or rolled into the required form. During the hot-rolling process, the scale falls through the roll tables into a trough or sewer through which rapidly flowing water is passing. The hot-rolled mill scale used in this work was provided by the mill of ALWEFAK IRON AND STEEL Co, SAUDI ARABIA. At first, the hot-rolled mill scale was dried in the furnace at 220°C to remove the moisture content before crushing and milling. The hot-rolled mill scale was manually crushed and milled to powder with size less than 75 micrometers using a sieve with 200 mesh. Figure 1a-f represents a block diagram showing the experimental setup representation and the tools used to compact and measure iron powder flow rate and density. Some of the produced specimens after compacting are shown in Figure 1g. The iron powder (50 g) is mixed with 5% kerosene and then pressed and compacted under a pressure force of 100 MPa. The green



**Figure 1.** Experimental setup representation; (a) Agate, (b) Electronic balance 4-digits, (c) Beakers with iron powder, (d) Beakers for measuring the shaking density of IP, (e) Hydraulic press, (f) Measuring IP flow rate, (g) Produced specimens by powder metallurgy.

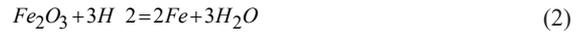
and sintered densities measurement of compacted and sintered specimens are obtained by Archimedes method. Porosities of the composites are calculated from the difference between the measured and theoretical density of each sample. The chemical analyses and the phases percentages comprising the hot-rolled mill scale powder were determined using XRD measurements (Bruker D8 advance diffractometer with a Cu-tube operated at 40 KV and 40 mA). The chemical composition of hot-rolled mill scale and produced iron powder were investigated by an Energy-dispersive X-ray fluorescence (EDXRF) spectrometer, Thermo Fisher Scientific, (model: Quant' X ) using Uniquant Standardless method and the free powder technique. The ground sample was placed in a pre-assembled plastic cup (40 mm diameter × 38.4 mm height) and covered with polypropylene support thin films of 3.6µm thickness (Chemplex industries Inco).

## 2.2. Reduction process

The reduction process was carried out in a tube furnace with a controlled gas atmosphere. There are entry and exit ports for gas inflow and outflow at the furnace. The temperature was controlled by thermocouple type K. The mill-scale powders were burdened in steel ladle, and therefore, the ladle was put in the tube furnace with a maximum temperature of 1200°C. The amount of the gases entered to the furnace was controlled by flow meters. At the initial time, the interior air in the furnace should be removed before each experiment. At the start of the reduction process, the hydrogen gas (H<sub>2</sub>) was blown into the system to remove the oxygen. After about 10 minutes, the process

started in the presence of hydrogen gas. The hydrogen flow rate was 0.6 l/min in all reduction processes. The hydrogen passed through the tube furnace system, then directed to the exit gases port, and was ignited for security reasons. The temperature in the furnace was gradually raised to reach the desired temperature. The reduction was performed within the temperature range of 800-1000 °C and maintained constant to ±10°C. The holding time at the maximum temperature was at least 2 hours. The process was then finished, and the furnace was turned off. When the temperature in the furnace fell under 100°C, the flow of the hydrogen gas was stopped until the furnace temperature was totally cooled down. Afterward, the steel ladle was withdrawn from the furnace. A schematic representation of the reduction procedure with the used tools and equipment are shown in Figure 2a-i. As observed in this figure, the drying, crushing, and sieving of mill scale briquettes are shown in Figure 1a-d, and the tube furnace used is also shown in Figure 2e. From Figure 2f-i, the preparation of the produced iron powder is observed after the reduction procedure at different temperatures.

The hot-rolled mill scales contain three types of iron oxides. Their reduction with hydrogen gas (H<sub>2</sub>) occurs according to three basic reactions:



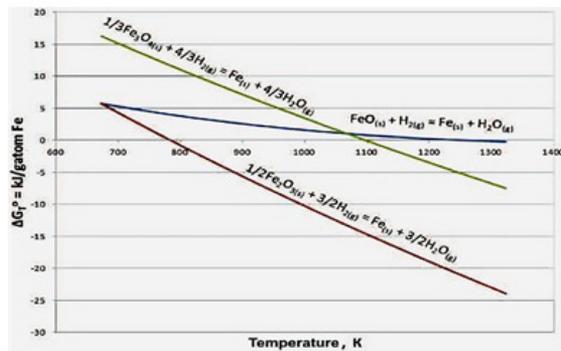
**Figure 2.** Schematic representation of the reduction procedure with used tools and equipment's; (a) Mill scale, (b) Mill scale after drying at 250°C, (c) Hand mill, (d) Sieving process, (e) Tube furnace with H<sub>2</sub> gas system, (f) Produced iron powder, (g), (h), (i) preparation of produced iron powder.

Figure 3 shows the temperature sequence of the isobaric-isothermal potentials (Gibbs free energy) of the above reactions 1, 2, 3 (reduction with the hydrogen of the iron oxides). Figure 3 also displays that  $\text{Fe}_2\text{O}_3$  is with the greatest reducibility. It was reduced to metallic iron powder at temperatures above  $500^\circ\text{C}$  (773 K). The temperature of the hydrogen reduction of  $\text{Fe}_3\text{O}_4$  is higher, about  $827^\circ\text{C}$  (1100 K).  $\text{FeO}$  is the most resistant for the reduction, especially at lower temperatures. It is reduced to about  $980^\circ\text{C}$  (1250 K). Since the rolled mill scale is a mixture of these iron oxides, the reduction temperature with hydrogen has to be in the highest temperature interval  $980\text{--}1000^\circ\text{C}$ <sup>13</sup>.

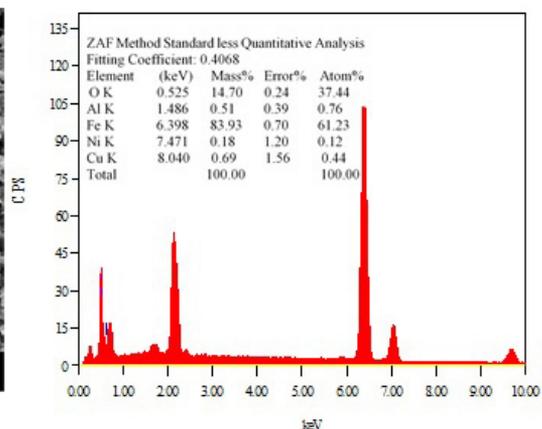
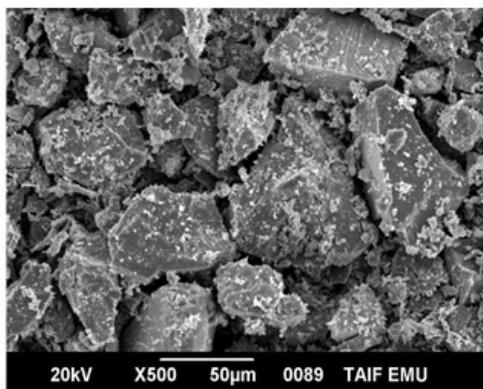
### 3. Results and Discussion

#### 3.1. Characterization of rolling mill scale

Figure 3 shows SEM image and correspondent EDS spectrum analysis of hot-rolled mill scale. The morphology



**Figure 3.** The changes of the Gibbs free energy with the temperature of the reactions of reduction with hydrogen  $\text{H}_2$  of the iron oxides (mill scales)<sup>13</sup>.



**Figure 4.** SEM image and correspondent “EDS” spectrum analysis of Hot-Rolled mill scale.

**Table 1.** Average chemical composition of the tested hot-rolled mill scale.

Element	O	Fe	Mn	Ca	Ti	Cr	Cu	Ni	K	Zr	Re
Mass %	30.029	69.37	0.310	0.111	0.0623	0.0350	0.0278	0.0129	0.0129	0.0078	0.0077

of the mill scale is mostly particulates with a surface formed basically by a matrix of iron oxides, as shown in Figure 4a. EDS spectrum analysis of mill scale and corresponding elements composition are given in Figure 4b. Table 1 shows the chemical composition of the elements concentration in the hot-rolled mill scale. The chemical composition of the hot-rolled mill scale was investigated by Energy-dispersive X-ray fluorescence (EDXRF) spectrometer, which depends on the sample composition before the reduction process. Figure 5 presents the result of qualitative XRD peaks’ profile and phase analysis of the used hot-rolled mill scale powder (with the size down to 200 mesh) before the reduction process. This figure shows that the hot-rolled mill scale is composed mainly of hematite ( $\text{Fe}_2\text{O}_3$ ) and magnetite ( $\text{Fe}_3\text{O}_4$ ). The chemical analyses and the phases percentages comprising the hot-rolled mill scale powder were determined using XRD measurements (Bruker D8 advance diffractometer with a Cu-tube operated at 40 KV and 40 mA). It can be seen that the dominant phases are hematite ( $\text{Fe}_2\text{O}_3$ ) and magnetite ( $\text{Fe}_3\text{O}_4$ ), while the amount of Fe metallic is insignificant. Under this condition, the Fe phase is still dominantly the oxide-based phase, typical of rolled mill-scale, as confirmed by EDS analysis.

#### 3.2. Characterization of rolling mill scale after reduction process

##### 3.2.1. Metallographic Analysis

Milled mill scale reduction experiments were carried out in a hydrogen atmosphere furnace at temperatures of 800, 900, and  $1000^\circ\text{C}$  for 2 hours holding. Figures 6-11 illustrate SEM images and corresponding “EDS” analyses and X-Ray analyses of the reduced rolled mill scale at different temperatures (800-1000 $^\circ\text{C}$ ) in a hydrogen atmosphere. The spectrum of the elemental composition of the iron powder produced after reduction at 800, 900, 1000  $^\circ\text{C}$  are shown in Figure 6b, Figure 8b, and Figure 10b, respectively,

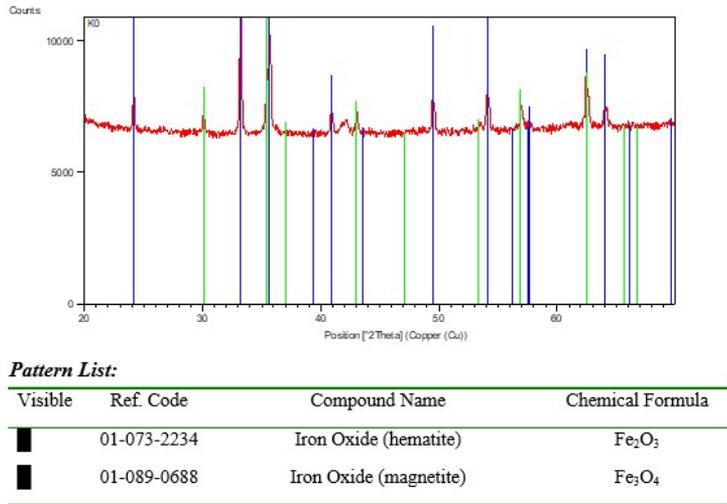


Figure 5. Result of qualitative XRD analysis of the used Hot-Rolled mill scale powder.

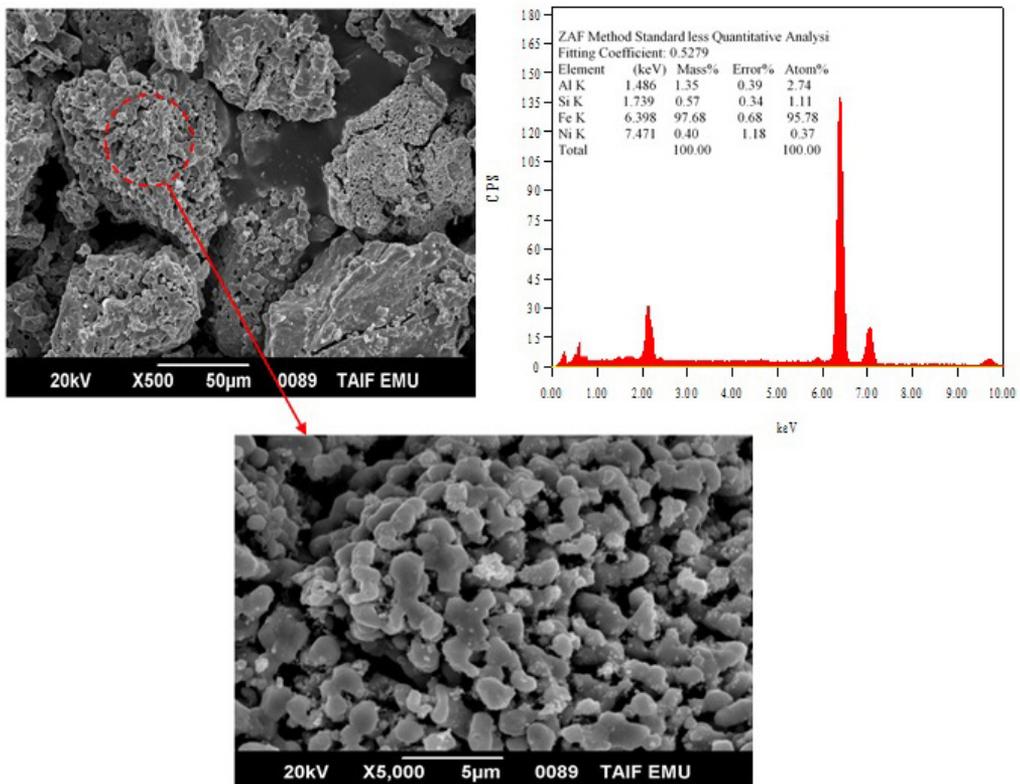


Figure 6. SEM image and correspondent “EDS” spectrum analysis of reduced iron powder at 800 °C in H<sub>2</sub> atmosphere.

in hydrogen gas (H<sub>2</sub>). This confirms that the amount of the reduced iron powder increases with the increase of reduction temperature. The X-ray diffraction (XRD) spectra of the reduced iron powder at different temperatures of 800, 900, 1000 °C are shown in Figure 7, Figure 9, and Figure 11, respectively. These figures show that the sharp peaks of

iron (Fe) are observed with a small peak of Mn, Zn, Mg, and Na elements. From which it is clear that the reduction at high temperature (1000 °C) more iron is produced and reached about 98.58%. Figure 10a shows a powder with a spongy appearance, irregular, with a high specific surface area that makes it highly suitable for powder metallurgy

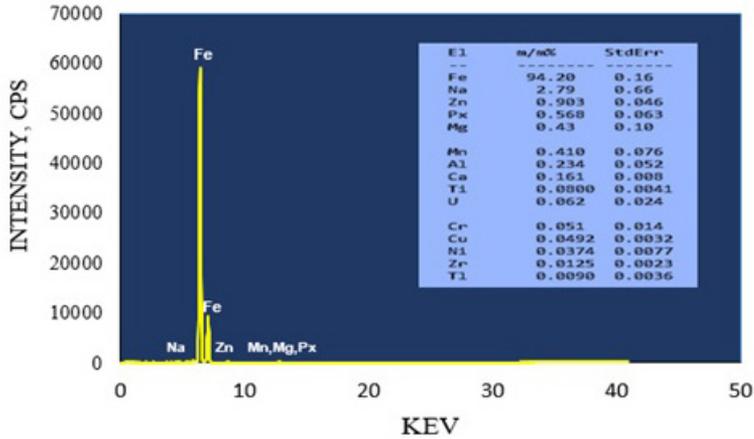


Figure 7. The X-Ray spectrum analysis of elemental composition of the iron powder produced after reduction at 800 °C.

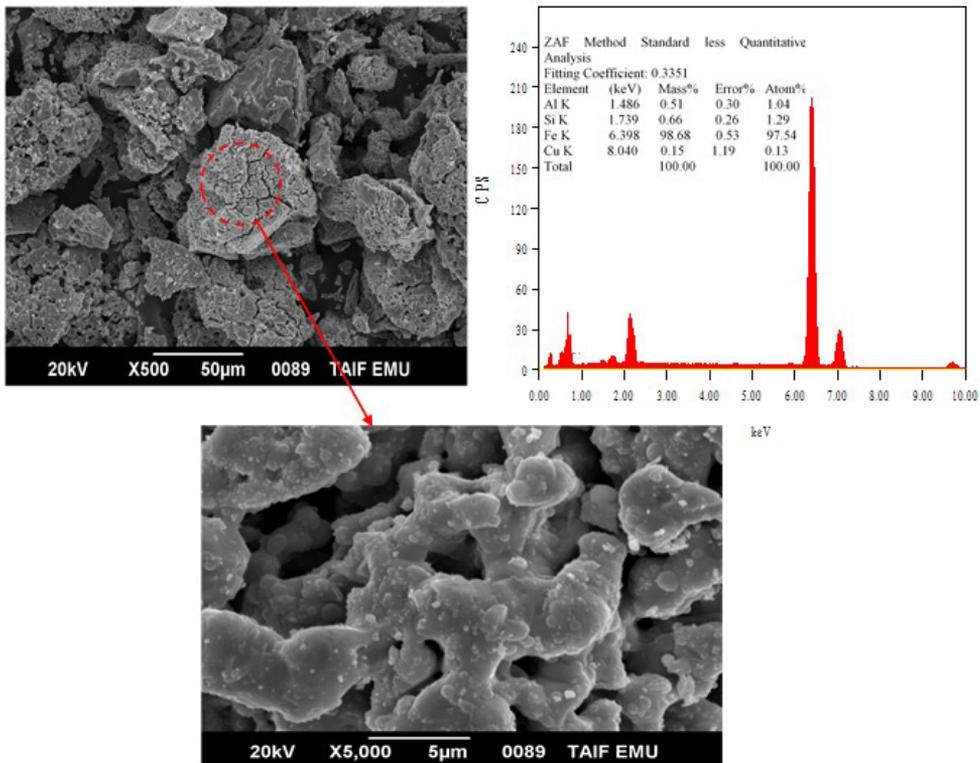


Figure 8. SEM image and correspondent “EDS” spectrum analysis of reduced iron powder at 900 °C in H<sub>2</sub> atmosphere.

applications. The oxygen content in these samples was 0%. Figure 11 shows the X-Ray analysis of the mill scale at 1000°C after reduction in the H<sub>2</sub> atmosphere furnace. The XRD peaks profile of corresponding metallic iron is observed, which confirms that in this case, the reduction of the mill scale to sponge iron powder has been completed. The obtained iron powder can be used to produce powder metallurgy parts by pressing and sintering because its size distribution can assure a good flow behavior.

Figure 8 shows SEM image of the mill scale after reduction at 900°C and corresponding “EDS” analyses. These analyses confirm the existence of metallic iron powder in most zones of the mill scale samples reduced at 900°C. XRD results for the reduction of mill scale with hydrogen at 900°C are shown in Figure 9. As can be seen, the metallic iron powder (Fe) produced was less than metallic iron (Fe) produced at 1000°C. Where metallic iron (Fe) produced at 1000°C was 98.58% Fe and metallic iron (Fe) produced at 900°C was

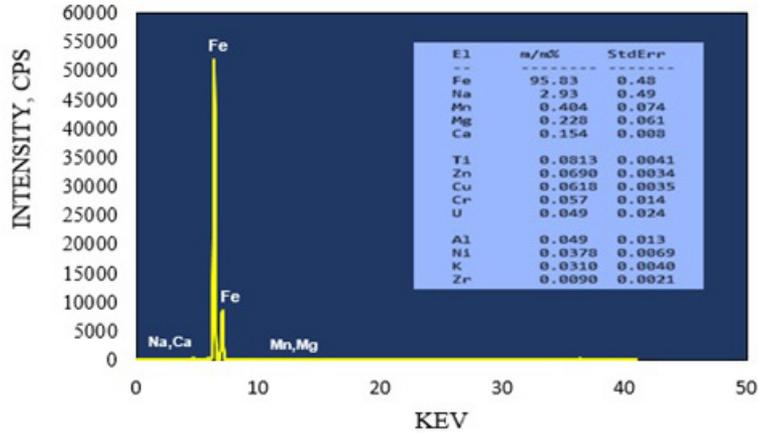


Figure 9. The X-Ray spectrum analysis of the elemental composition of the iron powder produced after reduction at 900 °C.

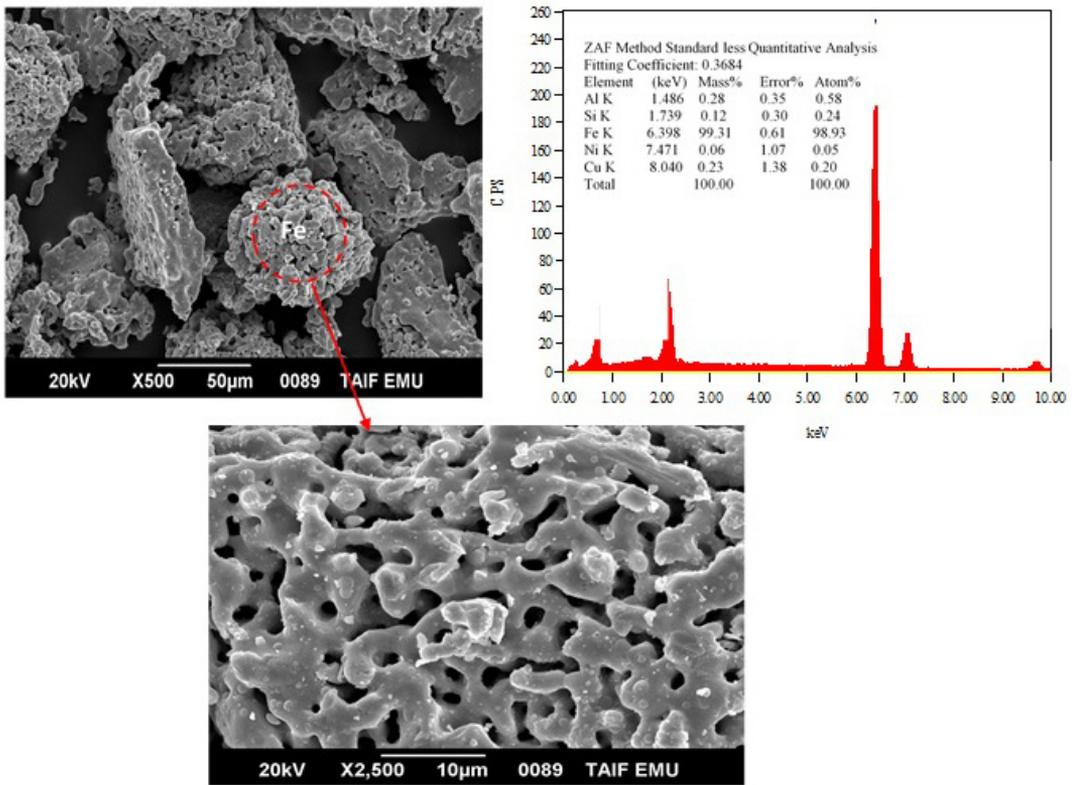
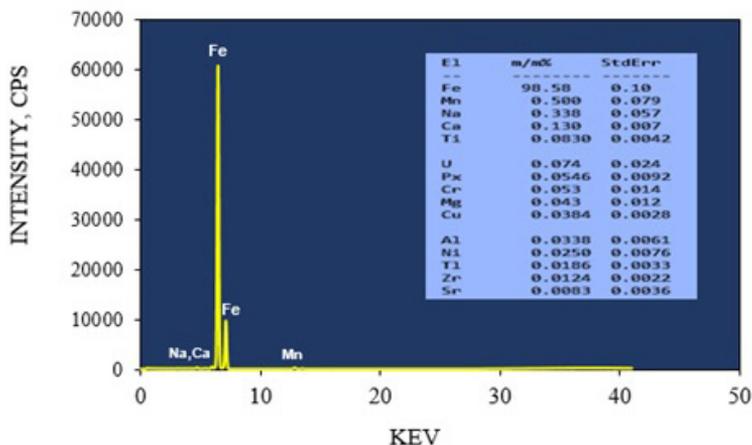


Figure 10. SEM image and correspondent “EDS” spectrum analysis of reduced iron powder at 1000 °C in H<sub>2</sub> atmosphere.

95.83% Fe. Figure 6a shows SEM image of a reduced mill scale at 800°C shows a powder with a spongy appearance, rounded with crystallite size less than the crystallite size at both 900 and 1000°C. XRD results for the reduction of mill scale with hydrogen at 800°C are shown in Figure 7. Where metallic iron powder (Fe) produced at this temperature, i.e., 94.20% Fe, was less than that produced at both 900 and 1000°C. At high reduction temperatures (more than 900°C), the oxygen removal favorable increased<sup>14</sup>. Wagner et al.<sup>15</sup> reported that

(i) most of the reaction features are very similar to that of the reduction by carbon monoxide and many mechanisms are common to both of them, (ii) the reduction with hydrogen is endothermic, whereas it is exothermic with carbon monoxide and conversely, thermodynamics is more favorable with hydrogen than with carbon monoxide above 800°C, (iii) with hydrogen, the hot gas-fed has to bring enough calories to heat and maintain the solid at a temperature sufficiently high for the reaction to occur, (iv) kinetics are also faster with hydrogen.



**Figure 11.** The X-Ray spectrum analysis of the elemental composition of the iron powder produced after reduction at 1000°C.

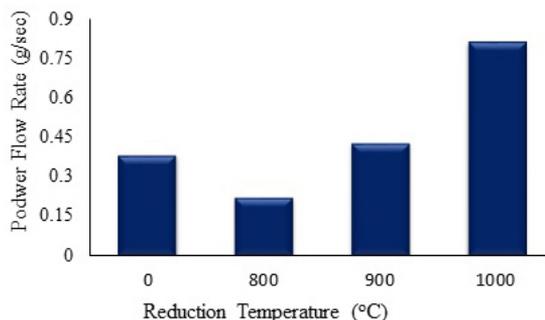
Scherrer equation, in X-ray diffraction and crystallography, is used in the determination of the size of crystals in the form of powder. Scherer's formula was used for the calculation of crystallite sizes:

$$\tau = \frac{K \lambda}{\beta \cos \theta} \quad (4)$$

where  $\tau$  is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the particle size,  $K$  is a dimensionless shape factor, with a value close to unity (about 0.9),  $\lambda$  is the X-ray wavelength used,  $\beta$  the broadening of diffraction line measured as half of its maximum intensity and  $\theta$  is the corresponding angle. Based on Equation 1, the estimated crystallite sizes of hot-rolled mill scale and reduced iron powder at different temperatures of 800, 900, 1000°C were found to be ranged from 44.9 to 60.38 nm, as shown in Table 2. It is clear that the lower crystallite size of 44.92 nm was obtained for the produced iron powder after reduction at a temperature of 800°C, while the higher value of the crystallite size of 60.38 nm was observed for the reduced iron powder at a reduction temperature of 1000°C as indicated in Table 2.

### 3.3. Effect of reduction temperature on the produced iron powder flow rate and density

A method of determining the flow behavior of a metal powder has been described by adding a small quantity of iron powder, e.g., 20 g at a time to a tube having a cone end with an orifice of 2.5mm into which the powder would fall. The orifice of the cone was sealed by means of a metal disc. The iron powder was allowed to fall freely throughout the cone end orifice, and the time was recorded. The flow rate of the reduced iron powder was calculated by dividing the known amount of iron powder by the elapsed time. Figure 12 illustrates the effect of temperature on the powder flow rate of a reduced mill scale at different temperatures (800-1000°C) in a hydrogen atmosphere. From this figure, it is clear that the powder flow rate increased as temperature increased. This may be attributed to the increased particle size with increasing temperature. The increase in temperature leads



**Figure 12.** Flow rate (gram/sec) of iron powder produced at different reduction temperatures.

**Table 2.** Crystallite Size (nm) of reduced iron powder at different reduction temperatures.

Reduction temperature (°C)	Crystallite Size (nm)
0	55.063
800	44.915
900	56.41
1000	60.38

to the raise of hydrogen adsorption, and subsequently, the rate of reaction increases, and the reduction ion increases<sup>16,17</sup>.

Also, the higher flow rate prevailing in the reaction zone enhances the rate of hydrogen absorption and, subsequently, the rate of chemical reaction steps increases<sup>18</sup>. The increase of reduction percentage with the rise of temperature may be due to the increase of the number of reacting moles having excess energy, which leads to an increase in the reduction rate<sup>16,19</sup>. In addition, the rise of temperature leads to an increase in the rate of mass transfer of the diffusion and rate of desorption<sup>17-20</sup>.

The apparent density of a powder is the mass of a bulk volume of the powder. It is a density of a metal powder after it has been shaken down or packed by tapping or vibration. It is important in packing, storage, or transport

of commercial powders and in pressing practice where dies are vibrated to consolidate the die fill. The apparent density of iron powder was measured by allowing a graduated glass cylinder of specified size containing a known weight of iron powder to fall freely and normally onto the table, and the volume of the powder was determined by direct observation. The apparent density before and after shaking was calculated by dividing the known amount of iron powder by its volume. Figure 13 shows the density of iron powder produced at different reduction temperatures before and after shaking. This figure shows that all the apparent powder densities after shaking are higher than the densities before shaking for mill-scale and reduced iron powders at different reduction temperatures. This may be due to the lower porosities of the powder after shaking. In addition, higher apparent densities before and after shaking were observed for the reduced iron powder at 800°C, and lower values were observed for the reduced iron powder at 1000°C.

### 3.4. Effect of sintering on density of compacted iron powder

Figure 14 shows the measured green and sintered densities of the compacted iron powder, produced at different

reduction temperatures of 800, 900, and 1000°C in hydrogen gas, before and after sintering at 1000°C, respectively. This figure shows that the higher densities in both green compacted and sintered conditions were recorded for the reduced iron powder at 800°C. At the same time, the lower values of the green compacted and sintered densities were observed for the reduced iron powder at a reduction temperature of 1000°C. This may be due to the lower crystallite size of the reduced iron powder after reduction at 800°C that equals 44.92 nm. The higher crystallite size value of the reduced iron powder after reduction at 1000°C equals 60.38 nm. The powder characteristics have an important influence on the green and sintered densities. One of the advantages of powder metallurgy is that the density of sintered parts is easy to adjust by regulating the processing variables such as compacting pressure, sintering temperature, and time<sup>21</sup>. The compaction pressure has an important influence on the extent of pore closure and weight gain. The green and sintered densities increase with increasing compaction pressure and significantly influence the powder structural characteristics. Sintered density varies linearly with the green density (density before sintering) at the same compacting pressure<sup>21</sup>. The density of compacted specimen after sintering

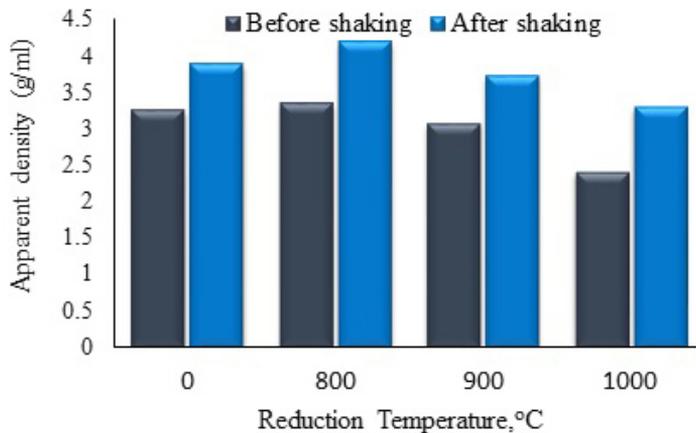


Figure 13. Measured density of iron powder produced at different reduction temperatures before and after shaking.

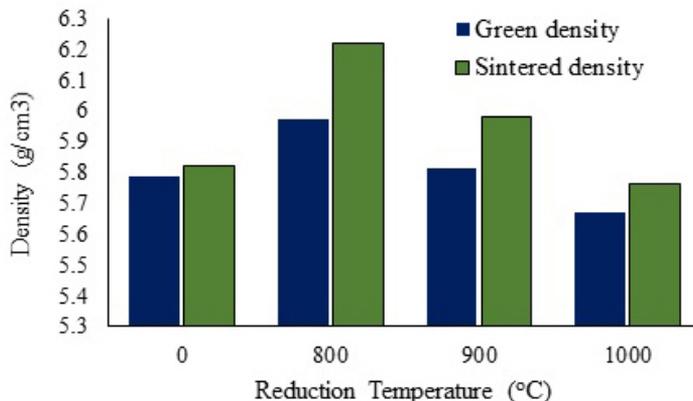
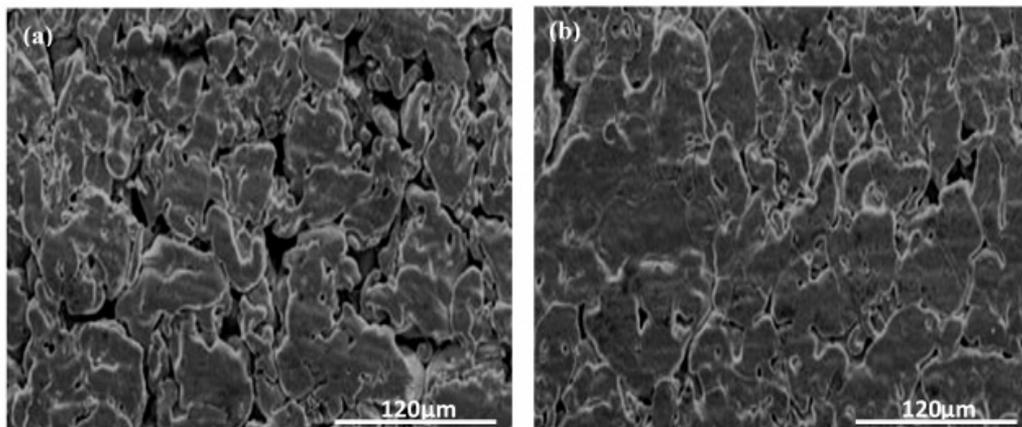


Figure 14. Measured green and sintered densities of the compacted iron powder produced at different reduction temperatures before and after sintering at 1000°C, respectively.



**Figure 15.** Surface morphology of sintered sponge iron powder (NC) specimens; (a) compacted at 300 MPa, (c) compacted at 600 MPa.

at a temperature of 1000°C for 2 hours is higher than that before sintering for all compacted iron powders reduced at different temperatures of 800, 900, and 1000°C in hydrogen. Figure 15a, b shows SEM images of the surface morphology of NC compacts with pressures of 300 and 600 MPa and sintered at 1140°C for 40 minutes<sup>21</sup>. Figure 15a exhibits many large pores with the compacting pressure of 300 MPa, but lower porosity was observed with compacting pressure of 600 MPa as shown in Figure 15b.

#### 4. Conclusion

- The hot-rolled mill scale is one of the most valuable industrial waste because of its iron content (in the range of 70-72% Fe).
- The reduction rate of hot-rolled mill scale briquettes/powder under a constant flow rate of hydrogen increased with the increase of the process temperature.
- The most effective reduction process in the presence of hydrogen gas atmosphere had been those carried out at 1000°C, at the holding time of 2 hours. The amount of iron powder produced at 800°C is 94.55%, but it is 98.22% at the reduction temperature of 1000°C.
- The proposed reduction process allows the obtaining of a sponge iron powder of an irregular morphology with different crystallite sizes as indicated by Scherrer equation, where the crystallite size increased as the reduction temperature. The minimum crystallite size value was 44.45nm at the reduction temperature of 800°C, but it equals 60.1nm at 1000°C.
- The produced iron powder obtained after the reduction process may be used in manufacturing and conventional steel-making processes or as a high purity raw material in powder metallurgy processes.

#### 5. Acknowledgement

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