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Hydrogen Reduction of Mill Scale for Iron Powder Production

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Abstract. This study explores the hydrogen reduction of industrial mill scale as a method for obtaining iron powder suitable for powder metallurgy applications. Mill scale, a by-product of hot rolling, was subjected to reduction in a hydrogen atmosphere at 800 °C, 900 °C and 1000 °C for 60 minutes under isothermal conditions. The chemical composition of the obtained products was analyzed using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). The results demonstrate that reduction at 800 °C produces sponge iron with approximately 95 wt.% Fe, accompanied by residual oxygen and calcium. At 900 °C, variations in impurity distribution were observed, reflecting the heterogeneous structure of the raw scale. The highest degree of reduction occurred at 1000 °C, where the iron content exceeded 98 wt.% and no harmful impurities such as sulfur or phosphorus were detected, although localized oxide inclusions remained. The findings confirm the feasibility of recycling mill scale through hydrogen reduction, providing both ecological benefits and economic advantages by converting metallurgical waste into valuable feedstock for powder metallurgy.

Keywords: iron powder, hydrogen reduction, mill scale, waste recycling, powder metallurgy.

Introduction

Mill scale, a solid by-product generated during hot rolling of steel, is composed mainly of iron oxides - hematite (Fe_2O_3), magnetite (Fe_3O_4), and wüstite (FeO) - with a total iron content of up to 70–75%. Despite this, it is often treated as waste, resulting in large-scale storage and associated environmental concerns. Recycling mill scale as a raw material for powder metallurgy aligns with the principles of sustainable metallurgy and the circular economy [1–3].

Hydrogen metallurgy is considered a promising alternative to conventional carbon-based reduction methods. Unlike carbonaceous reductants that introduce impurities and generate CO_2 emissions, hydrogen allows for the production of high-purity metallic iron with significantly lower environmental impact [4, 5]. The thermodynamics of hydrogen reduction are favorable at elevated temperatures, as the equilibrium constants of endothermic reactions increase with temperature, thereby enhancing reduction efficiency [6].

Recent studies have shown the potential of hydrogen reduction in laboratory and pilot-scale conditions. Han and Lee [7] emphasized that the kinetics of hydrogen reduction improve considerably with temperature. Zhang et al. [8] highlighted hydrogen metallurgy as a viable low-carbon pathway for steelmaking. Iwase et al. [9] demonstrated successful hydrogen-based reduction of Fe-containing oxides [10] for powder metallurgy applications [11]. However, research directly targeting the recycling of industrial mill scale into iron powder remains limited, leaving a technological gap.

The aim of this study is to evaluate the reduction behavior of mill scale under hydrogen atmospheres at 800–1000 °C, to analyze the composition of the reduced powders, and to assess the feasibility of this process for industrial powder metallurgy.

1. Materials and Methods

The material used in this study was iron scale, obtained as a by-product of steel production. The scale was received in powder form and mainly consisted of iron oxides. Before the experiments, the powder was carefully prepared and placed in ceramic boats for reduction.

The reduction process was performed in a tubular furnace under a continuous flow of high-purity hydrogen (99.999%). Three different temperatures were selected for the experiments: 800, 900, and 1000 °C. At each temperature, the sample was held isothermally for 60 minutes to ensure sufficient reduction time. The height of the powder layer in the boats was kept at approximately 10 mm to provide uniform heating and effective gas–solid contact.

After reduction, the samples were cooled inside the furnace in a hydrogen atmosphere to avoid secondary oxidation. The obtained powders were then subjected to analysis. Their chemical composition and phase distribution were determined using scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDX), which allowed both bulk analysis and point microanalysis of selected particles [12].

2. Results and Discussion

The initial scale was reduced at three temperatures: 800, 900 and 1000 °C in the hydrogen environment with the same isothermal holding time of 60 minutes. The height of the powder scale filling in the boat was 10 mm [13]. Figure 1 shows the appearance of the iron sponge made of scale reduced by hydrogen at 800 °C. An assessment of its chemical composition based on energy-dispersive analysis data is given in Table 1.

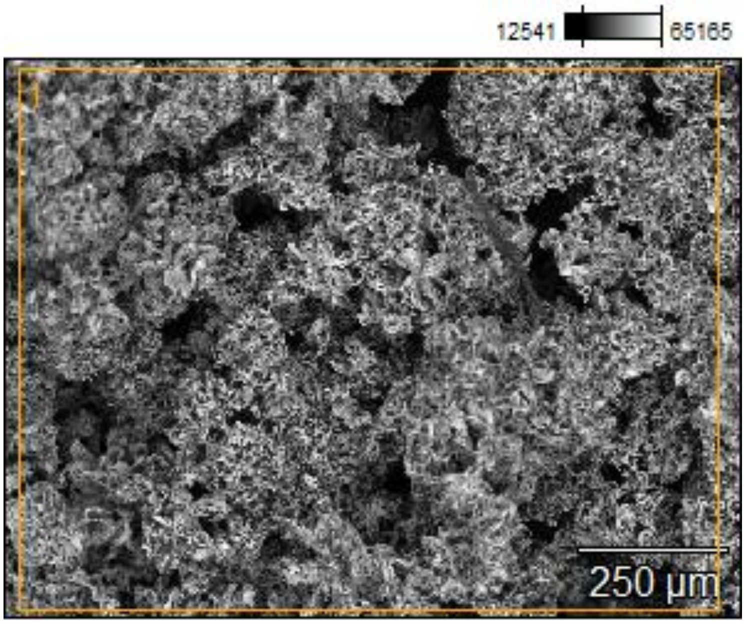


Fig.1. – Iron sponge made of scale reduced by hydrogen at 800 °C

Table 1. Elemental composition of iron sponge made of scale reduced by hydrogen at 800 °C

Element	O	Si	Ca	Mn	Fe
Content, wt. %	1.2	0.5	1.2	1.3	95.8

It can be seen from the presented results that the reduced iron powder has a fairly high content (total) of oxygen and calcium. At the same time, no harmful impurities of sulfur and phosphorus were found in it. Manganese was also found in the composition.

After reduction, the sponge was quite strong (from the point of view of its transportation for subsequent treatment), however, it was easily ground in a rotating ball mill [14].

The results of scale reduction at 900 °C are shown in Figures 2 and 3 (appearance) and in Tables 2 and 3 (chemical composition assessment).

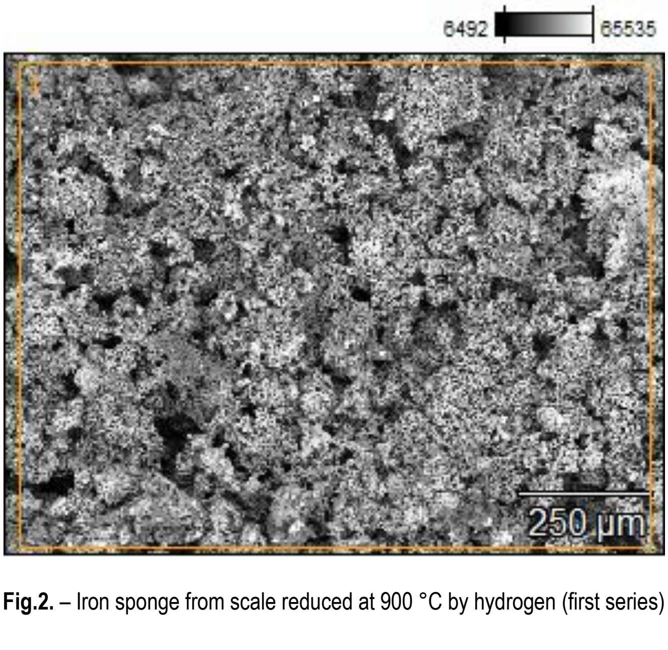


Fig.2. – Iron sponge from scale reduced at 900 °C by hydrogen (first series)

Table 2. Elemental composition of the iron sponge made of scale by oxygen reduction at 900 °C (first series)

Element	O	Si	S	Ca	V	Fe	Ni
Content, wt. %	2.1	0.6	0.5	1.3	4.4	89.8	1.2

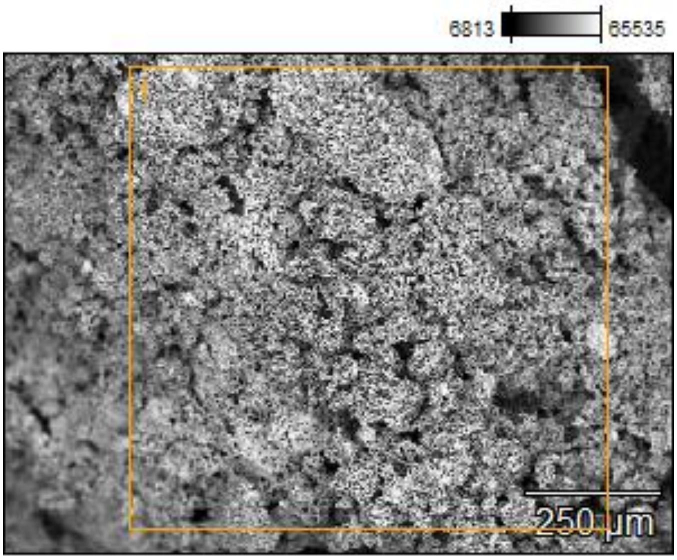


Fig.3. – Iron sponge from scale reduced at 900 °C by hydrogen (second series)

Table 3. Elemental composition of iron sponge obtained using scale reduced by hydrogen at 900 °C (second series)

Element	O	Si	Ca	Mn	Fe
Content, wt. %	1.5	0.6	1.4	1.2	95.3

After scale reduction at 900 °C in the first series, the amount of oxygen and calcium remained virtually unchanged compared to scale reduction at 800 °C. However, in this case, sulfur, vanadium, and nickel were detected in the absence of manganese. The reason for such discrepancies can be that the specified by the fact that impurity elements are present as a separate phase, distributed non-uniformly in the total volume of the scale [15].

This assumption is confirmed by the second series of experiments to determine the chemical composition of the iron powder made of scale reduced at 900 °C. From Table 4 it follows that sulfur, vanadium, and nickel were no longer detected in the powder under study but manganese was detected again. It should be noted that the amount of calcium remained virtually unchanged.

The appearance of the powder obtained using scale of the Qarmet JSC reduced at 1000 °C is shown in Figures 4 and 4. The corresponding results of the chemical composition assessment are presented in Tables 4-6.

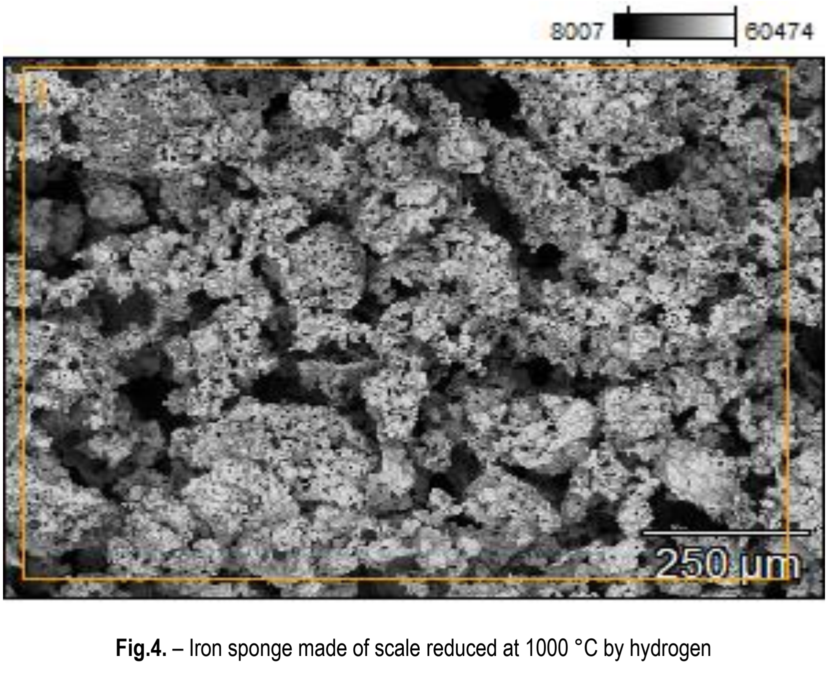


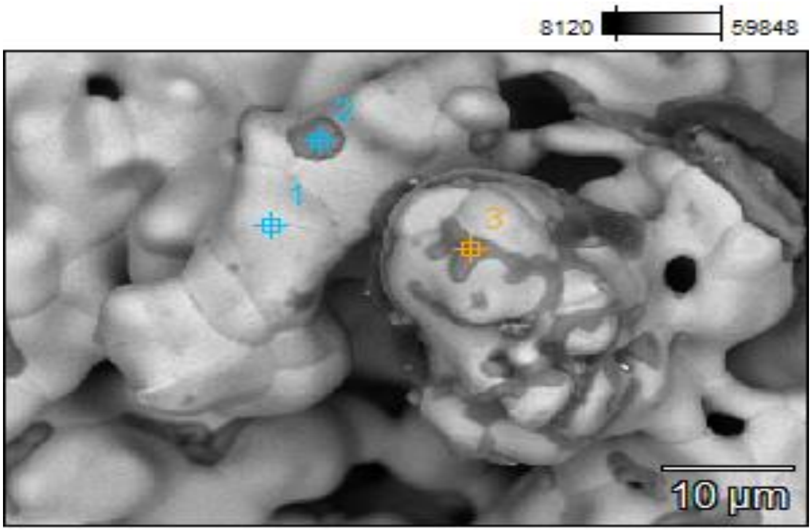
Fig.4. – Iron sponge made of scale reduced at 1000 °C by hydrogen

Table 4. Elemental composition of the iron powder reduced using scale at 1000 °C

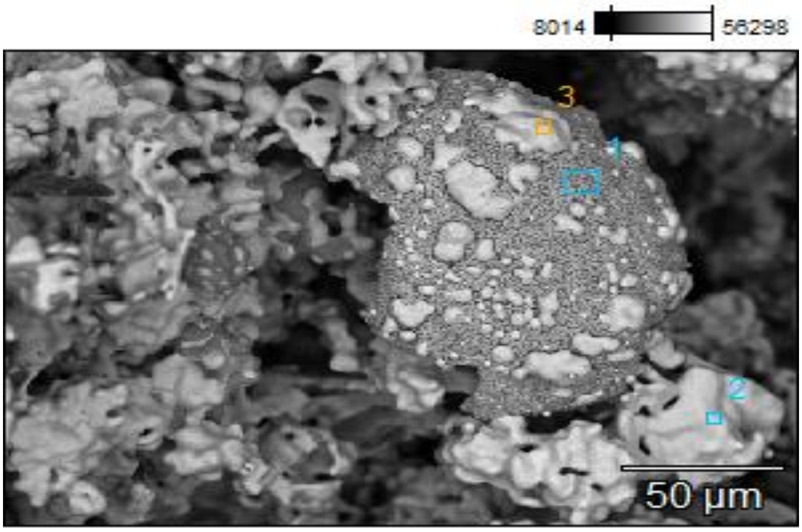
Element	Si	Ca	Fe
Content, wt. %	0.3	0.8	98.8

A general assessment of the chemical composition of a relatively large number of particles of iron powder reduced using scale did not show the presence of harmful impurities of phosphorus and sulfur of alloying metals [16]. At the same time, in addition to iron, silicon and calcium impurities were found in the powder.

For a more detailed study of the recovered powder, a point microanalysis of individual particles was carried out (Figure 5).



a)



b)

Fig.5. – Powder particles reduced using scale at 1000 °C

Table 5. Elemental composition of the powder particle reduced at 1000 °C using scale (Figure 6, a)

Point	Element	O	Na	Mg	Al	Si	P	Cl	Ca	Mn	Fe
1	Content. wt. %	-	-	-	-	-	-		-	-	100.0
2	Content. wt. %	37.4	0.7	-	0.3	12.0	4.5	-	39.2	2.7	3.1
3	Content. wt. %	32.8	1.3	0.6	-	11.3	1.2	0.4	28.0	1.7	22.7

Table 6. Elemental composition of the powder particle reduced at 1000 °C using scale (Figure 6, b)

Point	Element	O	Mg	Al	Si	Ca	Ti	Cr	Mn	Fe
1	Content. wt. %	0.5	0.8	1.3	2.1	3.8	2.3	24.3	19.6	45.4
2	Content. wt. %	-	-	-	0.3	0.8	-	-	-	98.8
3	Content. wt. %	-	-	-	-	0.8	-	3.0	2.2	93.9

The results show that the particles of the obtained powder were heterogeneous in their chemical composition. The reduced scale powder consists of several phases:

- pure iron (reduction up to 98.8%);
- a complex oxide phase based on iron and the other metallic elements [17];
- a complex oxide phase that does not contain iron.

Conclusions

Industrial mill scale, composed mainly of hematite, magnetite, and wüstite, is confirmed to be a promising secondary raw material for producing iron powder, reducing environmental problems associated with its disposal.

Direct hydrogen reduction at 800 °C resulted in partial conversion to metallic iron. The reduced powder contained residual oxygen and calcium, as well as manganese, indicating incomplete reduction at this temperature.

At 900 °C, variations in the chemical composition of the reduced powders were observed between experimental series. This effect was explained by the heterogeneous distribution of impurity phases within the mill scale. Nevertheless, no harmful phosphorus or sulfur impurities were detected in the final products.

The most efficient reduction occurred at 1000 °C, where the iron content exceeded 98 wt.% and the powders were free from sulfur and phosphorus. However, microanalysis revealed the persistence of oxide inclusions, showing that complete homogeneity was not achieved.

The reduced products exhibited satisfactory mechanical strength for handling and transportation, while remaining friable enough for subsequent milling, making them suitable for powder metallurgy applications.

The results highlight the technical feasibility of applying hydrogen metallurgy to recycle mill scale into high-purity iron powder. Compared with conventional carbonaceous reduction, the hydrogen route offers an environmentally friendly alternative with minimal emissions.

Further optimization, including refining of reduction parameters and potential post-treatment methods, may increase the homogeneity of the powders and broaden their applicability in advanced manufacturing processes.

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