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DETERMINING VOLUME CHANGES AND POROSITY FORMATION IN THE OXIDATION AND REDUCTION PROCESS IN THE ENERGY STORAGE UNIT OF IRON-AIR BATTERIES USING COMPUTER PROGRAMS

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Abstract: This research aimed to investigate the problems arising in the energy storage unit for iron air batteries. In particular, the coefficients of expansion and contraction at different temperatures observed when using iron foam materials in the energy storage unit when reduced with hydrogen gas. Reduction reactions conducted at temperatures ranging from 700 to 1000◦C under hydrogen and forming gas. The expansion and contraction process, which is one of the main problems encountered in the reduction of iron oxide with hydrogen and its use as an energy storage unit, has investigated. Microstructure and porosity of iron foam material used as energy storage unit analyzed. In this study, we examined the porosity structures formed during the reduction of iron oxides using two different reducing gases. The formation of microstructure and porosity and their amount calculated using computer programs.

Keywords: hydrogen reduction of iron oxides; iron oxides; swelling index; porosity, energy storage unit

1. Introduction

Currently, powder metallurgy is an industry undergoing rapid development. It encompasses the production of powders, metals, and alloys with particles of varying dispersion, granules, threads, and other forms. This industry focuses on manufacturing diverse machine parts, devices, tools, and specialized items. Additionally, it involves the development and creation of metallic powder materials along with non-metallic substrates. Powder metallurgy techniques also include the strengthening and restoration of machine parts and devices, as well as enhancing the mechanical or special physicochemical properties of product surfaces through coating with powder composite materials.

Among the various technological methods used for producing structural products, the cold pressing method in closed molds followed by sintering is widely employed. This approach offers high productivity and enables mass production with minimal mechanical processing, leading to reduced waste and metal losses by 10-15 times even for products with complex shapes. Moreover, this method presents endless possibilities for creating new materials based on iron powders using alloying and heat treatment techniques. [12]

In recent years, hot pressing technology has seen rapid advancements. This technique involves simultaneous pressing and sintering of powders, resulting in dense products with specific mechanical properties. Hot pressing allows for lower process temperatures, slowing down recrystallization processes and leading to fine-grained structures and improved mechanical properties in low-temperature ranges.

Although hot pressing methods require more complex equipment, special tooling preparation, and may have lower performance compared to conventional sintering, the benefits in terms of strength properties and wear resistance justify these differences from both technical and economic perspectives. Hot working with pressure methods is particularly effective for manufacturing large products using challenging-to-sinter or substandard powders, where high density and strength are necessary, surpassing what is achievable through conventional sintering processes. [13]

The residue left from hot rolling, known as mill scale, has yet to be widely explored for recycling and reuse by researchers and manufacturers. This study aims to convert mill scale into iron powder using hydrogen gas, contributing to environmental recycling and the creation of new functional porous materials from this by product. The reduction process of mill scale is achieved using a combination of hydrogen and an H2/N2 gas mixture.

The Uzbek Metallurgical Plant (Uzbekistan, Bekobod) produced 800 thousand tons of rolled steel per year. By 2024, it is planned to produce 1.04 million tons of rolled products [14]. During the rolling process, 1-3% of the finished product turns into scale. Only the Uzbek Metallurgical Plant collects about 20 thousand tons of mill scale.

Process analysis focuses on understanding the patterns that lead to the structure of processed iron powders, particularly the porous structure formed during iron oxide reduction in the solid state. This porous structure enables the creation of various catalysts and filters from the iron powders, offering diverse possibilities. Controlling the return modes allows for adjustments to the dimensions of this porosity. Additionally, increasing the iron oxide content leads to the reduction from higher oxides to lower oxides, as evidenced by previous research [2–4].

Fe_2O_3–Fe_3O_4–FeO_n–Fe.

When iron oxide powders are reduced with hydrogen, a certain volume change occurs. That is, there is a significant difference in the density of hematite (4.9-5.3 g/cm^3) and pure iron (7.874 g/cm^3). However, the formation of pores in the structure of iron oxides in the solid state with hydrogen is observed. A similar phenomenon is not observed during the regeneration of iron oxides in liquid phases.

Accurate assessment of shrinkage and expansion rates is difficult because the porosity of the structure compensates for the shrinkage of the sample due to the difference in densities. The following analyzes were performed by observing the expansion index of iron oxide upon reduction with hydrogen under a high temperature microscope.

The formation of porosity of reduced iron powders is influenced by many special factors. These include the size of the oxide powder, the composition of the reducing gas, the gas flow rate and, of course, the temperature. Several scientists have conducted research not on the reduction of mill scale, but on the reduction of iron ore with similar indicators using H_2. [7-10]

During the planning of the study, thermogravimetric and thermodynamic analysis of hot rolling scale reduction was studied in researches conducted by authors such as Chuan Guan. [21]

The purpose of this study is to analyze the swelling index and porosity parameters during the reduction of iron oxides formed during hot rolling in a volume of forming gas 80% N2 / 20% H2 and compare the results of reduction in pure H2 gas.

2. Experimental Section

2.1 Materials Used

Used pressed samples from powders of pure rolling scale from Uzbek Metallurgical Plant. Mill scale is purified by washing in process water, drying at a temperature of 250 °C, passing through a magnetic separator, flotation concentration and roasting (Table 1). The sizes of pressed powders are in the range of 100-1000 microns. The initial diameter of the sample is $2r_0$=6 mm. The cross-sectional surface of the pressed samples is in the range of 61.5 – 66.0 mm^2.

Chemical compositions of pure mill scale powders. Table 1

Elements	Fe total	Si	Al	$x \cdot 10^{-3}$											
				Ca	Mg	Mn	Ti	S	Cr	Ag	Cu	Pb	Ni	Co	Mo
Scale content, wt%	75,0	1,2	1,1	90	80	500	20	90	50	10	200	8	70	6	20

2.2 Methods

A high-temperature microscope, also known as a high-temperature tube furnace, was used to heat the pressed scale sample (Figure 1). By placing the sample on an Al_2O_3 ceramic substrate, a type B thermocouple was placed underneath it. One side of the furnace tubes was covered with quartz glass, and the other with a door adapted for linear movement of the thermocouple. Both sides are sealed with rubber gaskets. A DFK 51AUC03 Imaging source CCD camera mounted on the quartz mirror side. The sample was heated to a given temperature in a protective gas Ar (99.999%) with a flow rate of 2 l/min.

The heating rate is 10 °C/min. Once the furnace reached the set temperature, it was held at that temperature for 30 minutes. Then the argon supply was turned off and reflux was supplied. Pure gases H_2 and N_2 80%/H_2 20% were used as the reducing gas. In this study, the flow rate of reducing gases is 3 l/min^{-1}. The amount of gas consumed was measured with a flowmeter. A camera mounted on the side of the quartz glass was connected to a personal computer and the process was recorded on video. Reducing gas was supplied for 60 minutes. Then the H2 gas was turned off and Ar gas was introduced, and the furnace was cooled at a rate of 15 ^{o}C/min.

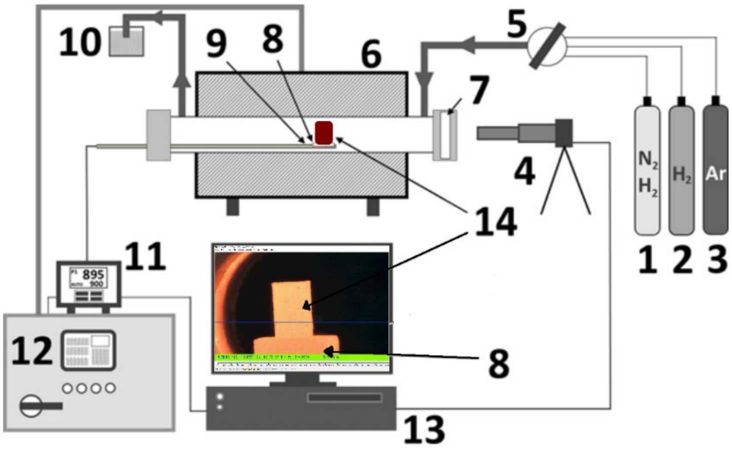


Figure 1. Device used to measure reduction swelling index (RSI) [5]

1) forming gas [N2/H2]; 2) hydrogen [H2]; 3) argon [Ar]; 4) charge-coupled device [CCD] camera; 5) gas inlet with gas flow meter (for H2, N2/H2, and Ar); 6) induction furnace; 7) quartz window; 8) ceramic substrate; 9) thermocouple Type B; 10) gas outlet with water bottle; 11) temperature controller; 12) generator for furnace heating; 13) PC; and 14) image of a iron oxide sample

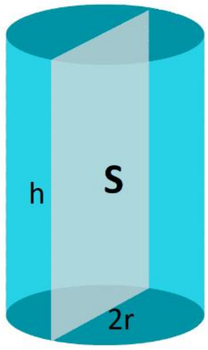
The images obtained during the experiment were processed in Stream Motion Software (Olympus, Japan) (Table 2). In this case, the cross-sectional area of the sample was measured through images taken by a camera oriented towards the operation of the furnace. In the first stage, changes in the cross-sectional area of the sample were measured every 10 seconds, and in the second stage, changes were measured every minute. The reason for this is that in the first minutes the recovery process is actively carried out in the H_2 environment. At the third stage, changes were analyzed every 5 minutes.

The following expression is used to calculate RSI:

$$RSI = (V_t - V_0)/V_0 \times 100\%$$

V_0 and V_t represent the initial and changed sample volumes per unit time, respectively. As we mentioned above, our samples are cylindrical in shape with a diameter of 6 mm. Using a high-temperature microscope, RSI was calculated by calculating the change in cross-sectional surface of cylindrical samples. In this case, the initial and temporary volumes of the cylinders are calculated using the following expression:

$$V_0=\pi r_0^2 h \qquad V_t=\pi r_t^2 h_t$$



As already mentioned, there is data on the scatter of cross-sectional areas of the samples. But to calculate this change in surface area, we need to determine the radius and height of the cylinder. We calculated the change in radius and height per unit time using the following expressions:

$$S_0 = 2r_0 \cdot h_0$$

$$S_t = 2r_t \cdot h_t \qquad k = \sqrt{\frac{S_t}{S_0}} \qquad r_t = r_0 \cdot k \qquad h_t = h_0 \cdot k$$

Here k is the proportional coefficient of change in cross-sectional area.

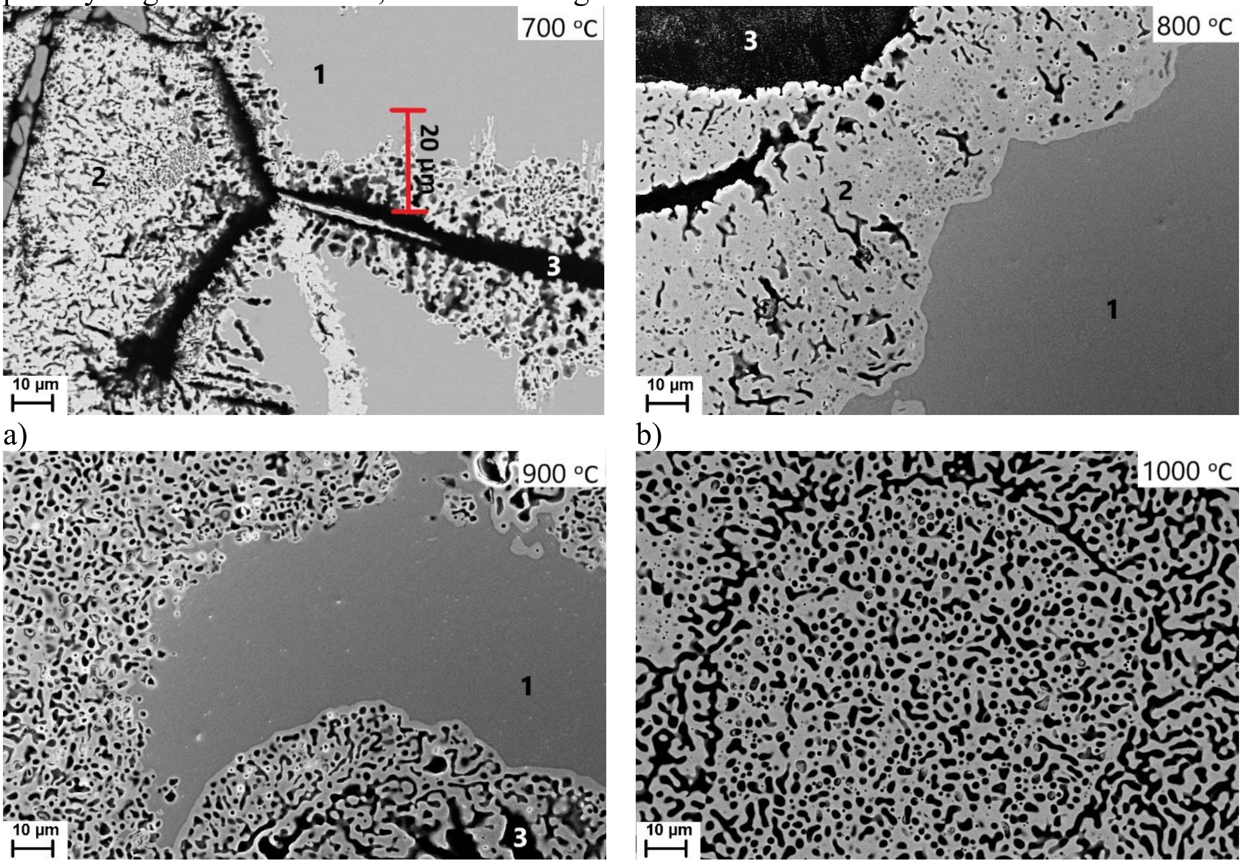
Microanalysis of the reduced samples was carried out using a scanning electron microscope SEM (Ultra55, Ziess NTS GmbH). The elemental composition of the phases was determined by energy-dispersive X-ray spectroscopy (EDS) on a microscope. For SEM microscopy, the samples were first embedded in epoxy resin, dried, and the surface of the samples was ground and polished.

3. Results and Discussion

3.1 Thickness of the reduced layer

The microstructures obtained from the experiments were scrutinized to determine the impact of the depth of oxide reduction, initial size of resulting oxide particles, the type of reduction medium (H_2; N_2/H_2), process duration, and temperature. To achieve this, studied the microstructure of samples that had been reduced in different modes, determined the size of the largest particles and measured the reduced layers on their surface based on the modes. The analyses carried out are outlined in Table 2.

A sample's microstructure, which was subjected to a 1-hour reduction at different temperatures and in pure hydrogen H2 conditions, is shown in Figure 2.



a) b)

c) d)

Figure 2. Microstructure of powder particles reduced in a hydrogen H_2 environment at 2000x.
1) Fe_2O_3 ; 2) Fe ; 3) epoxy

From Figure 2a shows that in an environment of pure H2 at a temperature of 700 °C, the size of the reduced layer in 1 hour was on average 10-20 µm. S.M.Shin, as shown in his experiments, at this temperature, the rolling scale cannot completely reduce to iron [16]. The reduced layer at a temperature of 800 °C - 20-40 µm (Fig. 2b), but under the same conditions. C.Joshi also experimented with the duration of scale reduction at such low temperatures and reported that the scale was not completely reduced [17].At a temperature of 900 °C, the thickness of the reduced layer was on average 50-100 µm (Fig. 2c). A. T. Turkdogan stated that the particle size of the reduced powder influences the degree of reduce [18]. At a temperature of 1000 °C the largest particle in the microstructure (1000 µm) was completely reduce, therefore the average size of the reduced layer at a temperature of 1000 °C was 500-600 µm (Fig. 2d). In experiments conducted by Damien Wagner, it can be seen that the reduction time is significantly through the use of nano powders [21].

In Figure 3 shows the microstructure of samples reduced at different temperatures in an atmosphere of forming gas (80 vol.% N_2 / 20 vol.% H_2).

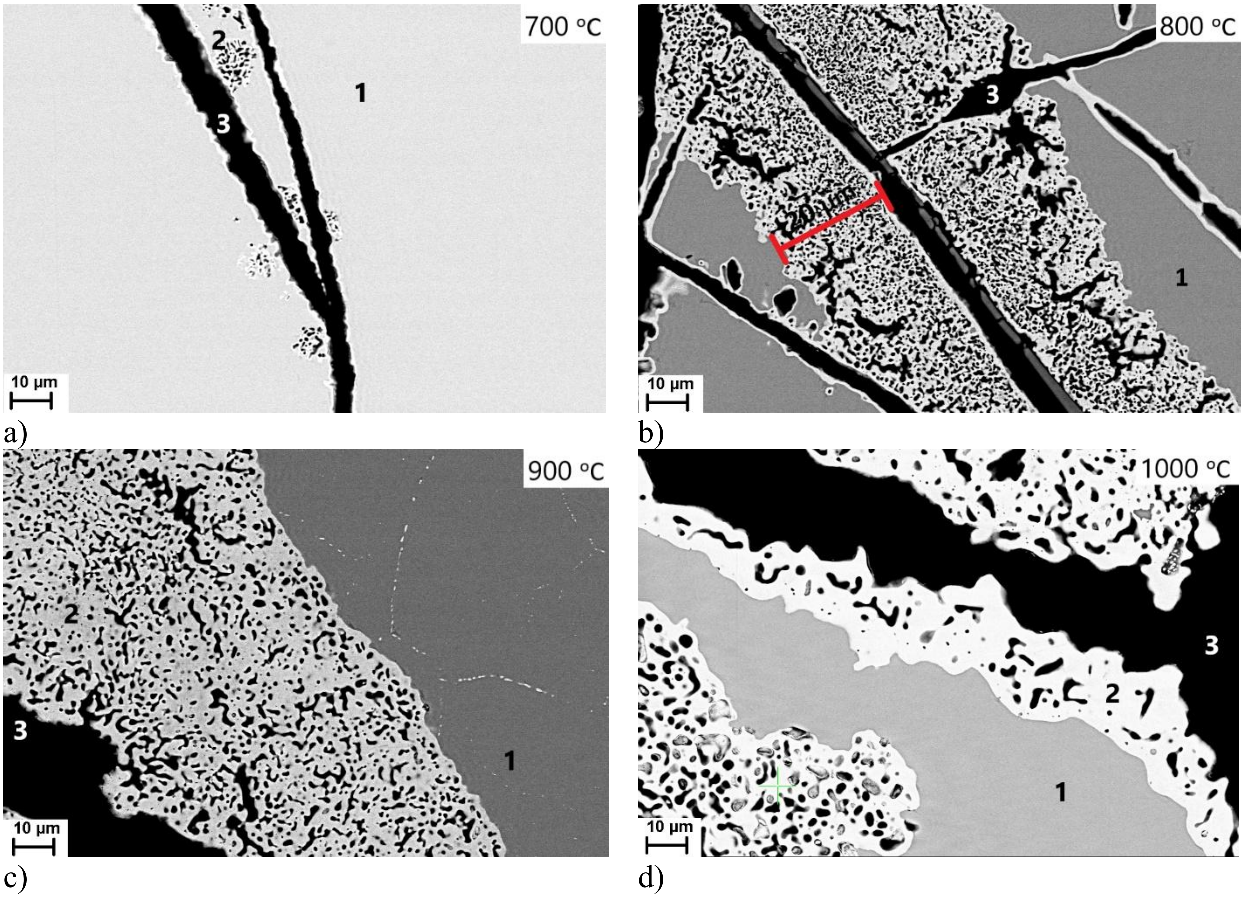


Figure 3. Microstructure of powder particles reduced in a forming gas (80 vol% N_2 / 20 vol% H_2) environment at 2000x.

1) Fe_2O_3 ; 2) Fe ; 3) epoxy

From Figure 3a it can be seen that the average size of the reduced layer was 2-5 µm in an forming gas environment at a temperature of 700°C for 1 hour, and the reduced layer was 10-20 µm at a temperature of 800°C. °C (Fig. 3b), but under the same conditions. The thickness of the reduced layer at a temperature of 900 °C was on average 40-50 µm (Fig. 3c), and in the microstructure of the sample reduced at a temperature of 1000 °C, the thickness of the reduced layer was 100-300 µm.

Effect of temperature, atmosphere and reduce time on thickness of the reduced layer. **Table 2.**

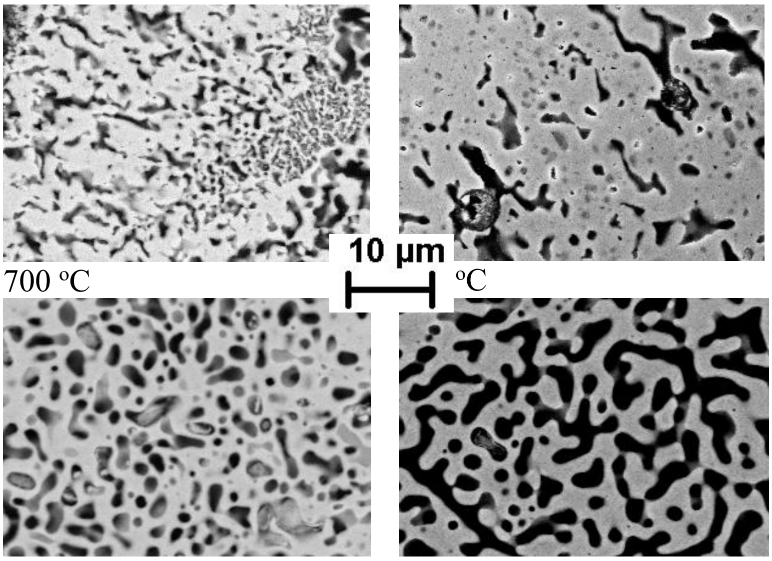
Thickness of the reduced layer **[µm]**	**Temperature [°C]**				**Time [hour]**
	700 °C	800 °C	900 °C	1000 °C	
Pure hydrogen H_2 environment	10-20 µm	20-40 µm	50-100 µm	>500 µm	1 hour
Forming gas (80 vol% N_2 / 20 vol% H_2) environment	2-5 µm	10-20 µm	40-50 µm	<300 µm	

In the above-mentioned microstructures, the contact surface of a single oxide particle with a reducing gas (pure hydrogen and forming gas) was analyzed. In the experiment, iron oxide particles with a size of 100-1000 microns were reduced at a temperature of 700-1000 °C.

From the microstructural analysis of samples selected in different modes and Table 2 filled out on its basis, it is clear that the reduction of oxides in both modes accelerates with increasing process temperature. At the same time, the rate of oxide reduction in an environment of pure hydrogen is on average 2 times higher than that of forming gas.

3.2 Pore size

In order to determine the influence of the reducing environment (H_2), time and temperature of the process on the porosity of particles of reduced iron powder, the microstructure of samples selected in different modes was determined. In Fig. Figure 4 shows the pore microstructure of iron particles regenerated in various modes in an environment of pure hydrogen H_2.



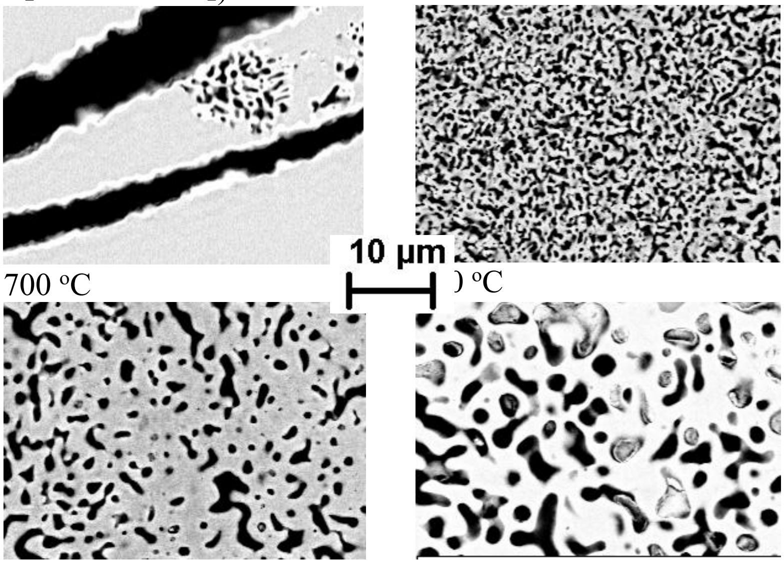
700 °C °C

900 °C 1000 °C

Figure 4. Microstructure of the porosity of a powder particle, reduced in a hydrogen environment H2 at 2000x.

From the microstructure in Figure 4 it is clear that an increase in the reduction temperature in a pure hydrogen environment (700-1000 °C) had a great influence on the shape of porosity and the particle size of the reduced iron. At relatively low temperatures, the pore size is small and the pores are not connected to each other. As the temperature increased, the pore size increased and as a result, the pores connected with each other. S. H. Kim provided detailed information on the effect of pore size based on his experiments on not only the reduction temperature but also the duration of the process [19].

To determine the influence of the reduction medium (forming gas N_2/H_2), time and temperature of the process on the porosity of particles of reduced iron powder, completely reduced particles were determined from the microstructure of samples taken in different modes. Only in the sample reduced at 700 °C were no completely reduced particles found. The reason, as we said above, is that the depth of the reduction is too shallow. In Fig. Figure 5 shows the pore microstructure of iron particles reduced in various modes in forming gas (80 vol.% N_2 / 20 vol.% H_2).



700 °C) °C

900 °C 1000 °C

Figure 5. Microstructure of the porosity of a powder particle, reduced in a forming gas (80 vol% N_2 / 20 vol% H_2) environment at 2000x.

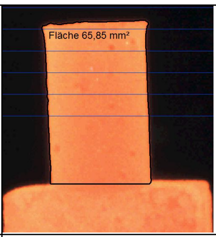
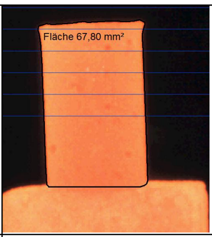
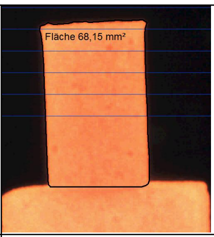
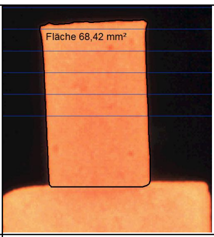
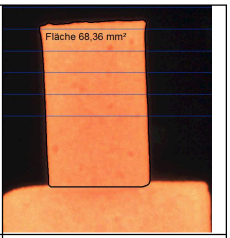
From the microstructure in Fig. Figure 5 shows that with an increase in the reduction temperature in the same gaseous environment (700-1000 °C), the shape of porosity and the size of the extracted iron particles have a significant impact. At relatively low temperatures, the pore size is small and the pores are not connected to each other. The pore size and shape of reduced iron

particles in samples taken in the forming gas, similar to those taken in pure hydrogen, changed with increasing temperature. From this we can conclude that the mechanism of reduction of iron oxide in hydrogen and forming gas environments is the same. O. Benchiheub conducted experiments on the reduction of scale using various forming gases and concluded that complete recovery of forming gases would not occur [20].

3.3 Reduction swelling index (RSI)

To determine the reduction swelling index, the cross-sectional surfaces of the samples were photographed and processed in Stream Motion Software (Olympus, Japan) during a transient recovery process in various environments and modes. Images of samples taken at different time intervals and their corresponding processing time intervals are shown in Table 3.

Image sequence processed by single-sample RSI calculation. Table 3.
(t=1000 °C, pure H_2)

OLYMPUS Cross-sectional area					
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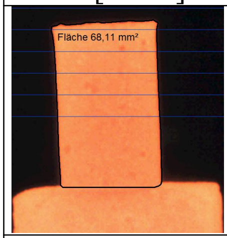	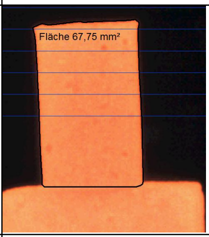	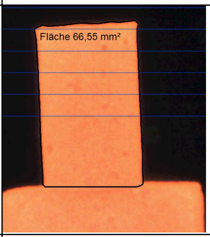	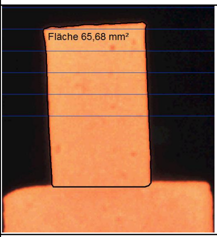	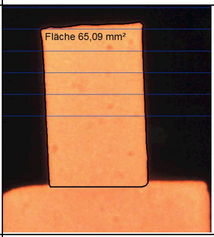	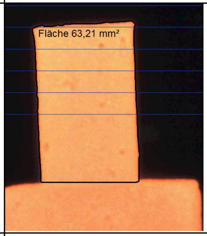
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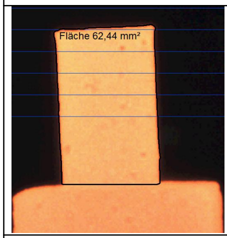	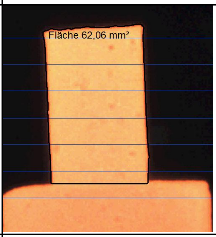	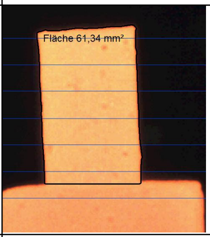	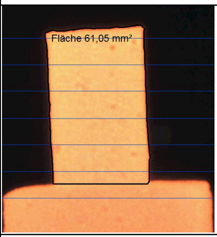	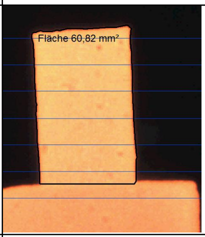	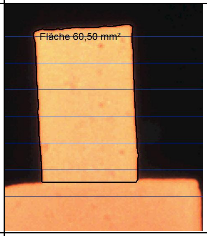
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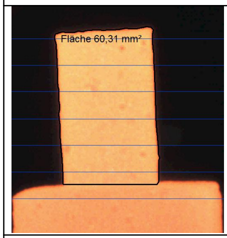	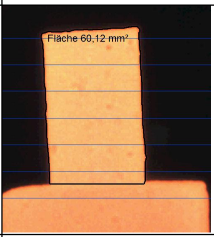	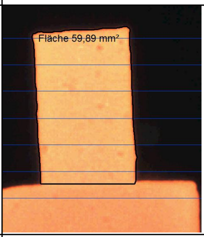	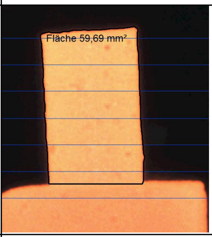	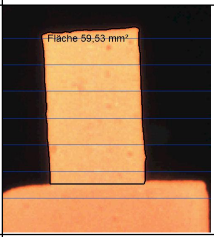	VCHI [%] Max = 5,91 % Min = -14,05 %
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Figure 6 shows the volume variation as a function of process time according to Table 3 using Stream Motion software.

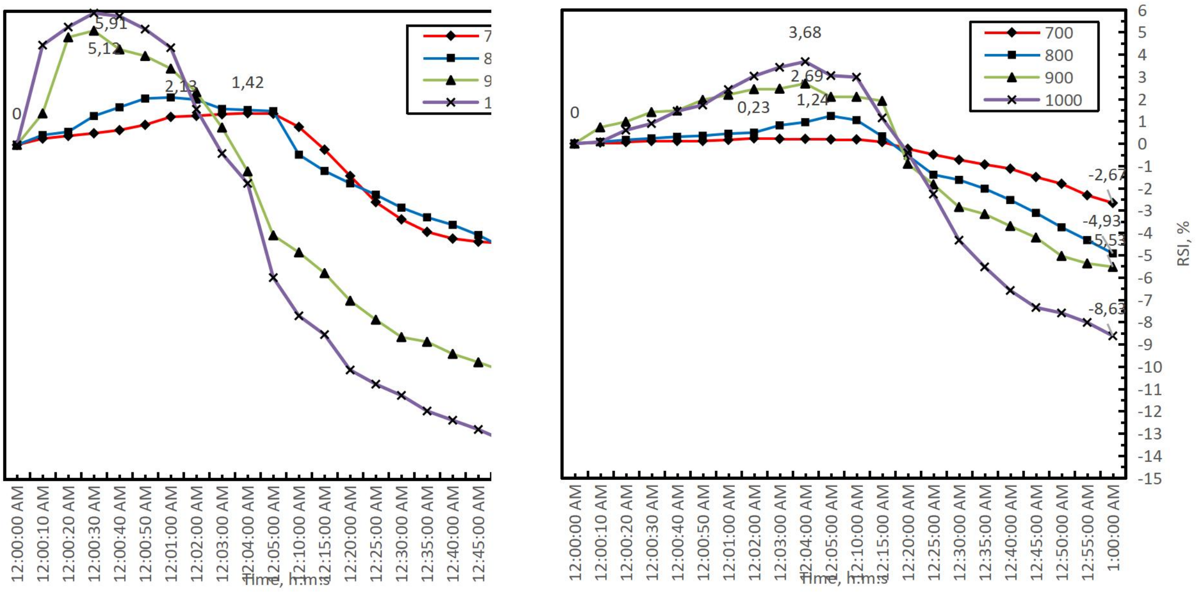

Figure 6. Change in the volume of a press briquette (iron oxide) reduced in various environments, temperatures and time intervals: a) hydrogen environment H2; b) forming gas (80 vol% N_2 / 20 vol% H_2) environment.

Based on the determined cross-sectional surfaces, the volume changes of the samples were calculated using the above expressions.

From Fig. 6a shows that during the first 3 minutes of the process in an environment of pure hydrogen, the volume of reduced samples increased to a maximum value at all temperatures. In our opinion, this is due to the adsorption of reducing gas molecules on the surface of oxide particles through the surface and pores of press briquettes. N. M. Gaballa deeply analyzed the interaction of reducing gases with the surface of the powder during the process of scale reduction, and the results of our research are also consistent with his conclusions [22]. The higher the temperature, the stronger the adsorption of the reducing gas on the surface of the sample particles. For example, the maximum swelling of a sample reduced at a temperature of 700 °C was 1.42%, at a temperature of 1000 °C - 5.91%, that is, adsorption at a temperature of 1000 °C occurs 4 times faster than at a temperature of 700 °C. After 3 minutes, the rate of hydrogen adsorption began to decrease compared to the rate of oxide reduction. As a result, the volume of the sample returned to its original state at the 4th minute. After 4 minutes of the process, the oxide sample was reduced in the pictures. In this case, the decrease in sample volume occurred due to the gradual loss of oxygen by the oxide. The higher the temperature, the smaller the initial volume of the sample. As a result, the porosity of the reduced sample increases (Fig. 4d).

By the 20th minute of the procedure, active narrowing can be seen. After 20 minutes you can see that the reduction swelling index has decreased. The result was that the sample returned from full oxide to metal within 20 minutes, with only massive plastic deformation occurring in the subsequent process. The reduction swelling index was -14.05%.

From the graph in Fig. 6b shows that due to the small amount of hydrogen in the first gas compared to nitrogen, the adsorption of hydrogen by the sample at a temperature of 1000 °C is small compared to pure hydrogen, and the reduction swelling index was 1.5 times smaller. The higher the temperature, the smaller the initial volume of the sample. Moreover, compared to pure hydrogen, the shape of the porosity formed in the sample was softer.

4. Conclusions

Before designing the microstructure of iron foam materials used as energy storage units (including laminar structures), it is necessary to take into account the volume change coefficients of iron oxide during hydrogen reduction and steam oxidation. In addition, regardless of the microstructural

architecture, the iron oxide particle forms pores throughout its grain during reduction. Research should be conducted on the use of other metal additives (eg Cs, Be, Mg, Sr, Ba) as ligatures to prevent the formation of these pores.

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