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Experimental Study on the Effect of Oxidation on the Compressibility of Iron Powder

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An experimental study was carried out on the effect of oxidation temperature and the oxide film composition on the compressibility of porous materials. Samples were annealed at different temperatures; the size change in the samples after annealing was measured. The phase composition of the oxide layer was investigated. Magnetite was generated at between 350 and 450°C, and two-phase oxide was formed at 550°C, after oxidation, weight gain was determined. The presence of pore overgrowth, which reduces porosity, was confirmed by metallographic tests. The maximum porosity is found in the oxidized samples produced by pressing at room temperature. The process of high-temperature oxidation of iron powder before pressing and in the state of free filling in a fluidized bed, as well as the effect of the content of oxides on magnetic characteristics, has been studied. The impact of oxidation on the compressibility of samples of iron powder was investigated. In this study, it was observed that the range of 350-450°C, offers the best compressibility and the necessary composition of the oxide film, which is also related to the presence of magnetite in the iron oxide coating. It is the ideal temperature for oxidation and repressing. The deformation of porous materials exposed to iron powder oxidation was tested.

Keywords: Oxidation, Iron Powder, Soft Magnetic Materials, Deformation, Porous Materials, Porosity, Compressibility, Magnetite, Pressing, Oxidation Temperatures, Iron Oxide Coating.

1 Introduction

Powder metallurgy is one of the processes used for producing metals and is commonly used. The high production quality and capacity to form pricey, very complex parts are what account for this widespread utilization. In order to produce a variety of products, this process is thus being used more frequently in place of a number of conventional metal processing processes [1]. In the process of powder metallurgy, metal or nonmetal powders are combined or blended, compressed into a mold to form the required geometry, and then sintered [2]. The term "powder" is used to describe solid particles that are smaller than 1mm in size and have extremely tiny characteristics [3,34].

Typically, metallic, powders have a low volume relative to their surface area, which is their most important characteristic [4]. Depending on the settings and processes used for producing the powder, each batch has its own unique qualities. It's crucial to comprehend the powder metallurgy processes used to create metallic powders in order to guarantee the quality of the material and powder. The powders' size, shape, and structure are just as crucial to powder manufacture as their chemical content and purity [5].

Any substance generally can be powdered. Atomization, chemical decomposition, and electrolysis are typically the three methods utilized to make powder. Atomization is a technique for crushing material by spraying it into gas or water as molten metal flows in a vertical or horizontal path [6]. The most important aspect of this method is the ability to control the shape and form of the particles.

Iron powder is manufactured in a variety of grades since each form of powder product is categorized into categories based on its purity, density, grain, and manufacturing process. As a result, we can get water-atomized iron powder for use in producing machine parts requiring strong components as well as light green strength powder for magnetic paints or printing inks [7].

Iron powder has a variety of applications that can be determined by the intended use. The following are only a few of the numerous varieties of applications:

Cars, trains, and airplanes. Materials and Products for Friction. For the necessary amount of friction, iron powder is widely used in brake pads, drum brake linings, and other applications. The iron powder used as a filler can increase the usefulness and durability of these objects by dispersing the generated heat [8,29]. Iron powder can be used to make sintered parts, goods, and components by mixing them with raw materials [9]

Another component that can be used to make soft magnetic composites is iron powder. These composites are subjected to heat treatment and compression to create isotropic components with

complicated shapes and three-dimensional magnetic properties [10]. Then, these soft magnetic composites are often employed in electromagnetic applications [11].

When brazing or welding is used to join the parts, iron powder is used to create a tight seal between the components [12]. Due to its capacity for high temperatures, the iron powder melts into a liquid flux and fills the space between the other two parts that need to be joined. Iron powder is sometimes used as a component of coating materials applied to coated welded electrodes or as a component of cored wires for welding [13].

Iron powder has been used as a surface coating for components that can be subjected to high temperatures due to its thermal abilities [14,31]. With this coating, components that have seen significant corrosion and wear will remain functional longer. The thermal surface coating could enhance the performance of the component or assembly.

Iron powder is also used in copy machines to make toner cartridges for color and black-and-white printers [15]. Because they are used to transport toner to the photoreceptor by charging it first, iron particles are included in carrier cores. The toner separates after being inserted during this electrophotography process [16]. In addition to the previously mentioned applications, iron powder can also be utilized in products like paints, dyes, oil filters, chemicals, and metal clays for jewelers.

It is well known that soft magnetic materials are combined in alternating magnetic fields using oxidized iron powder [17]. The physical and mechanical characteristics of sintered products are enhanced by the surface oxidation of metal powders [18]. The process for obtaining powders, their dispersion, the necessary features of products, etc. [19] all affect the oxygen content in powders, which offers the best combination of properties. It has been investigated how the iron powder is high-temperature oxidized before pressing and in the state of free filling in a fluidized bed, as well as how oxide content affects magnetic properties [20]. Compressibility is the proportional decrease in material thickness under predetermined conditions of increased pressure or compressive loading.

Because pressing causes significant particle deformation, particle hardness affects compressibility[21]. Additionally, some metals have harder working qualities than others. Nonmetallic substances like unreduced oxides have low specific gravity and high hardness, which both lower compressibility[22,33].

A powder's interior fine porosity tends to become unconnected during pressing, trapping air among the particles [23]. Air is incredibly compressible, but it doesn't take up much space and weighs very little. Powders that are nonporous are the most compressible [24,30]. Compressibility is the proportional decrease in material thickness under predetermined conditions of increased pressure or compressive loading.

Compressibility is frequently reduced by interstitial or substitutional hardening carried on by alloying additions or lingering impurities [25, 32]. Carbon has a much greater effect on compressibility than molybdenum, nickel, and manganese, which are intermediate elements for iron powders.

In particular, nickel and molybdenum very slightly reduce the compressibility of pre-alloyed steel powders, whereas chromium, copper, and manganese demand much greater compaction pressures [26]. In addition to the solid-solution strengthening, the significant oxidation propensity of chromium and manganese during powder manufacture is also blamed for the decrease in compressibility caused by these elements. The compressibility of iron powder is known to be reduced by residual oxygen.

2 Methodology

The aim of this work is to study the process of deformation of porous samples of iron powder subjected to oxidization. The study was carried out on commercially available iron powder grade FE-M-02-P without additional treatment [27]. A mixture was prepared by the addition of a 2% percent aqueous solution of polyvinyl alcohol. Samples had a mass of 40 grams.

To achieve 35, 30, and 25% porosities, 18 mm porous blanks were pushed at pressures of 200, 300, and 400 MPa. After pressing, the samples were heated for 0.80 hours at 850°C to drive out polyvinyl alcohol and water. Low temperature, short exposure, the influence of released water vapor, and gaseous decomposition byproducts of polyvinyl alcohol all impede shrinking processes.

Tab. 1 *Mechanical properties of iron powder FE-M-02-P*

Symbol	Fe
Molecular Weight	55.85
Appearance	Gray
Melting Point	1535 °C
Boiling Point	2750 °C
Heat of Vaporization	84.6 K-Cal/gm atom at 2750 °C
Poisson's Ratio	0.29
Specific Heat	0.106 Cal/g/K @ 25 °C
Thermal Expansion	(25 °C) 11.8 μm·m-1·K-1
Vickers Hardness	608 MPa
Young's Modulus	211 GPa

A caliper with an accuracy of 0.05 mm was used to measure the samples after annealing, and no change in size was found. Porous samples were subjected to vapor oxidation. To do this, they were placed in a container, loaded into an oven heated to 350, 450, 550°C, and kept for 10 min. in all temperatures. Through the inlet pipe, water vapor was pumped to the container which encouraged to oxidize for 30 minutes. An oxide layer formed on the surface of open pores and the sample's outer surface as a result of steam oxidation, according to the analysis of the microstructure of the oxidized samples. Visual inspection reveals that the film is dark, dense, and strongly bonded to the base metal.

The oxide film is equally distributed, according to a metallographic study that was done on samples that had not yet been etched and those that had been etched using a 5% solution of nitric acid in alcohol. The hardness of the oxide film is 800 MPa, which is slightly higher than the hardness of magnetite, while all samples have the same micro-hardness, which is equal to Hμ= 1200 across the grain body and equal to the hardness of ferrite.

Using the X-ray diffraction technique ADX-2700 θ-θ Powder X-ray Diffraction Instrument (XRD)[28] set up with filtered copper radiation and identical shooting circumstances, the phase composition of the oxide layer was investigated. Magnetite is generated between 350 and 450°C, and two-phase oxide is formed at 550°C, consisting of magnetite and wustite, according to calculations of the X-ray diffraction patterns of pressed samples that were oxidized under various conditions. It is clear why wustite appears at 550°C because based on the diagram of Fe-0 in its equilibrium condition, this temperature is where equilibrium exists. near the theoretical lower limit for forming wustite (570°C),

After oxidation, weight gain was determined as the sample's change in mass divided by their initial masses, $\Delta M/M_0$. Figure.1. shows the effect of initial porosity θ % and oxidation temperature on the weight growth ΔM/M0.%.

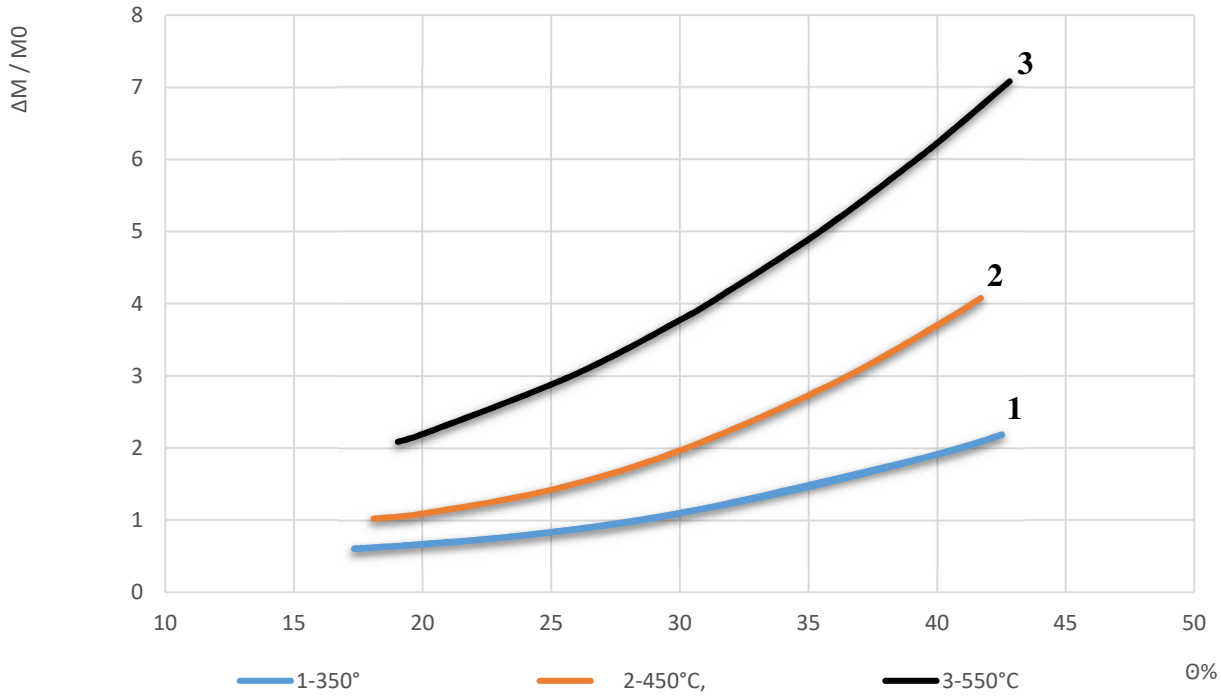

Fig. 1 *The effect of initial porosity θ % on the relative weight growth ⊿M/M0.%. at different oxidation temperatures 1-350°C, 2-450°C, 3-550°C*

3 Results and discussion

As porosity and oxidation temperature rise, the samples' mass rises as well. Oxidation caused a 12.4% average decrease in porosity. Metallographic experiments verified the existence of pore overgrowth, which lowers porosity. After oxidation, the samples were repeatedly pressed at an 800 MPa pressure both while still at the oxidation temperature and after cooling. The non-oxidized porous samples that had been sintered in a shielding media were again compressed to compare the compressibility. An indicator graphic called a "load-punch stroke" was captured while the object was deformed. The effect of pressure on the change in height during the pressing of samples is shown in Fig. 2. As shown, with an increase in the oxidation temperature of the samples, the compressibility deteriorates compared to non-oxidized samples.

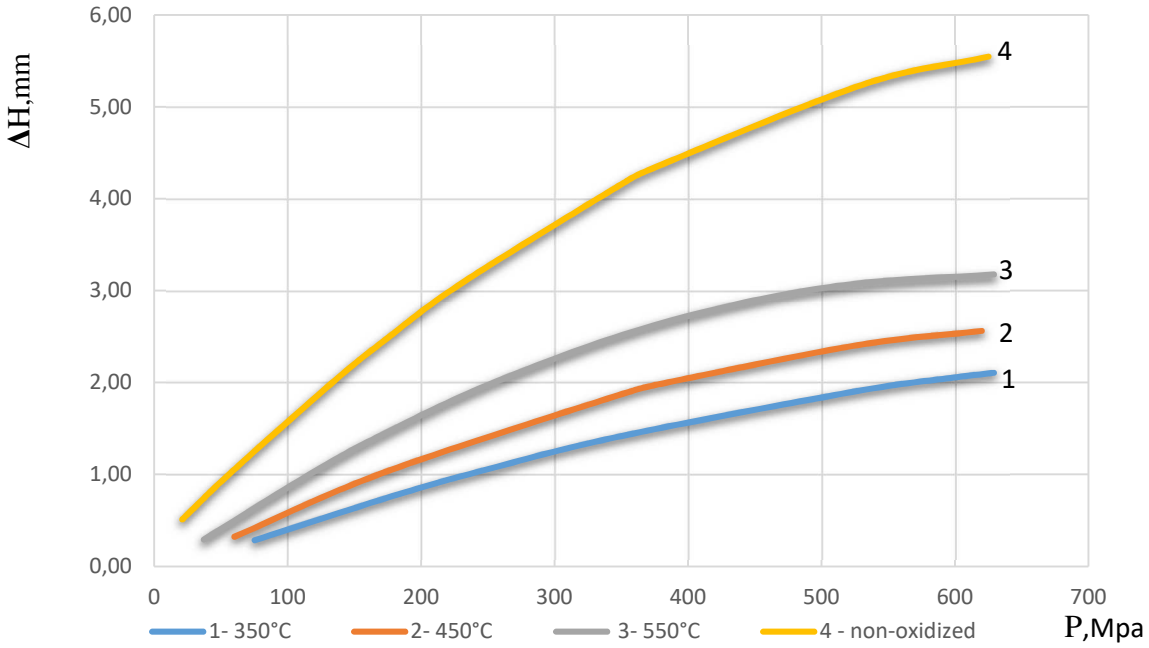

Fig. 2 *Effect of pressure on the height of oxidized samples 1-350°C, 2- 450°C, 3- 550°C, 4 - non-oxidized*

Figure 3 shows how the height of the oxidized samples differs from the non-oxidized sample. As can be seen, with increasing pressing pressure, the relative change in height decreases, and at an oxidation temperature of 350°C it is 30%, and at a temperature of type 550°C - 60% at a pressure of 600 MPa. Obviously, this is because magnetite has a lower coefficient of friction than a mixture of magnetite and wustite. As a result, as the temperature of oxidation rises, friction develops between the powder body's oxidized powder particles.

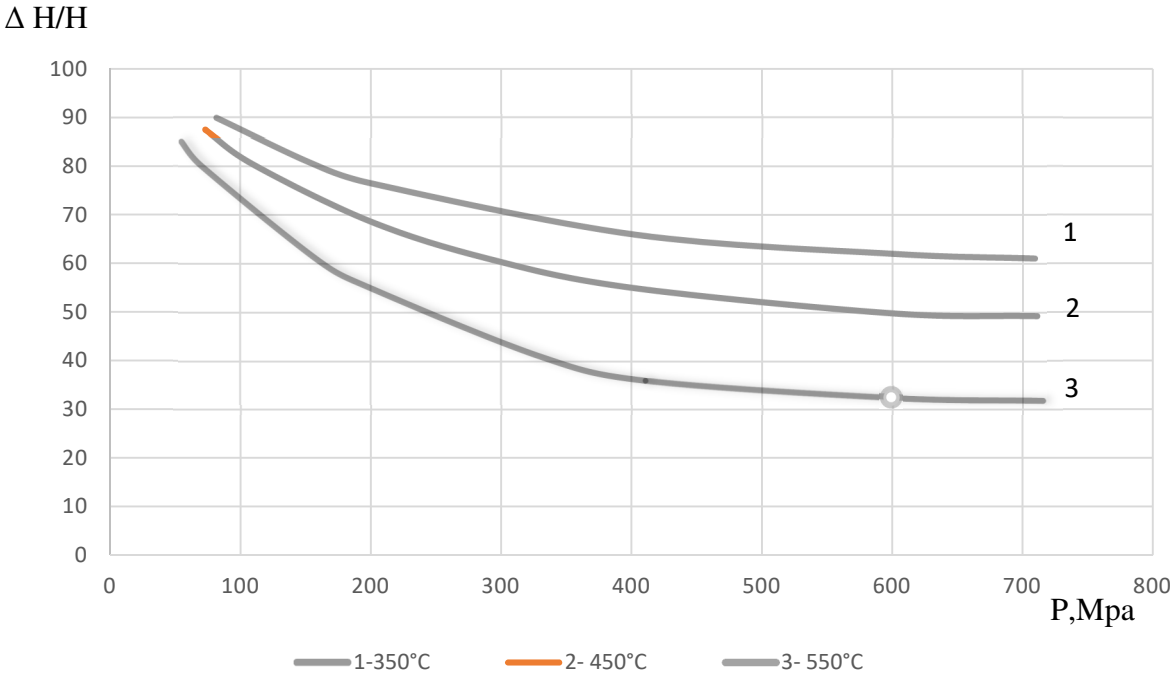

Fig. 3 *Change in the relative height of oxidized samples depending on the pressure of re-pressing 1-350°C, 2- 450°C, 3- 550°C*

Analyzing how the oxide deposit affects the change in porosity after repeated pressing is important. Figure. 4 illustrates the relationship between the initial porosity of the billet and the porosity after repeated pressing at temperatures right after oxidizing.

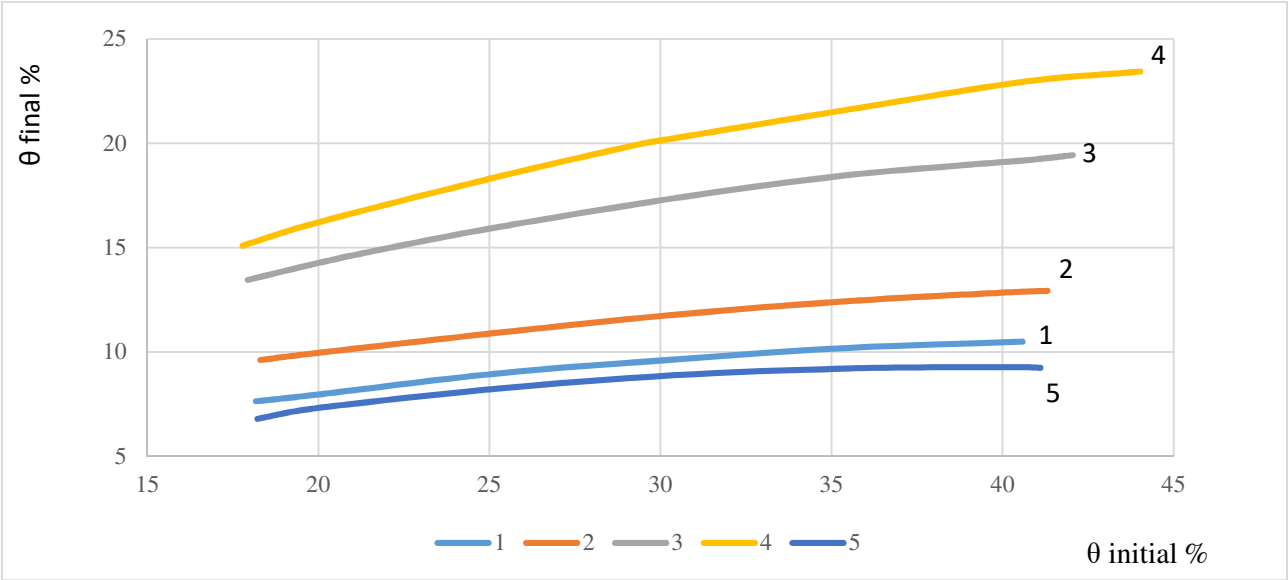

Fig. 4 *Change in porosity after repeated pressing at different temperatures: 1 - 350°C, 2 - 450°C, 3-550°C, 4-20°C, 5- non-oxidized 20°C*

4 Conclusions

The experimental results demonstrate that pressing samples with high porosity is difficult due to the oxide layers that develop on the samples after pressing.

The mass of the samples increases with increasing porosity and oxidation temperature.

It should be noted that in this instance, the difference in porosity between the oxidized and non-oxidized samples is negligible.

Since magnetite is produced at oxidation temperatures of 350–450°C, which increases the material's magnetic properties, these temperatures can be considered ideal for oxidation and subsequent pressing from the perspective of improving the material's magnetic properties.

It has been observed that temperatures between 350 and 450 °C, which are all related to the presence of magnetite in the iron oxide coating, should be taken into account when determining the ideal oxidation temperature at which sufficient compressibility is obtained.

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