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Sintering of Iron Powders by Resistance Heating Technique

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Highlights

- This article focuses on the efficient sintering method.
- A more efficient technique is proposed in the study compared to traditional sintering methods.
- It was concluded that it could be a highly effective and more efficient sintering technique.

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Abstract

In this study, powder metal samples of 15x15x4 mm dimensions of Fe powders were produced at different pressures (650,850,1050 MPa). They were sintered in atmospheric environment with the help of resistance sintering method applied for the first time using electric resistance welding machine. The sintered and green densities of the powder metal samples were determined and their sinterability in the unprotected environment was examined. In addition, the effects of electric resistance heating technique on sintering were investigated by performing hardness measurement and microscopic examination of sintered samples. As a conclusion, powder metal samples were sintered successfully with the electric resistance welding technique in an unprotected environment and high-density values such as 98% were obtained.

1. INTRODUCTION

Powder metallurgy is the process of pressing very small particles together and binding them into a whole. Nowadays, when cost and quality are the target, part production with powder metal techniques is becoming increasingly important. Importance of powder metallurgy; It is due to the fact that parts that are difficult or impossible to shape by casting, machining or plastic deformation can be produced easily and economically in mass production by this method [1]. Although small pieces produced by powder metallurgy were found in Egypt in 3000 BC, its first important use was in 1920 as the production of tungsten-carbide cutting tools. After the Second World War, it took its place in technology as a new part manufacturing technique in modern terms. With the powder metal technique, some pieces with a maximum weight of 45 kg and a length of 500 mm can be produced. 70% of powder metallurgy products are used in the automotive industry, 12% in construction machinery, 5% in agricultural equipment and 13% in existing tools. However, its use in the production of aircraft industry, high-tech composites, electronic components, magnetic materials and cutting tools used in machining is rapidly increasing. The powder metal market in the world is growing 12% a year. The most used metal powder is iron powder with 85%. Copper alloys come in second with 6-7% [2].

Powder metallurgy is well suited for mass production of small, complex and dimensionally sensitive parts. Material loss is very low. In this method, there are no melting losses, close tolerances and smooth surfaces are obtained. However, in addition to all these, the shape of the part to be made is a limiting factor due to the limited fluidity of the powders in the mould. In addition, the initial investment costs (presses, sintering equipment) are quite expensive. Depreciation values will be high if mass production is not made [3]. In this production method, pure metals, alloys, carbon, ceramics and plastic materials in the form of fine particles are formed under pressure by mixing with each other. Then these parts are sintered at a temperature below

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the melting temperature of the main component, creating a strong bond between the contact surfaces of the particles to achieve the desired properties. Among the metallic powder metal parts, the production of iron powder metal parts takes the first place. The reasons for this, fast, economical production and the ability to produce in desired sizes can be listed. Iron-based powder metal parts are mostly produced by using traditional (conventional) sintering method in industrial applications. Very long batch type furnaces are used in traditional sintering process. To perform the sintering process, heating these furnaces at desired temperatures and fixing them at sintering temperatures provides serious energy consumption and energy costs. Today, different sintering processes are used and tested to provide the same sintering process faster and cheaper. The leading ones are SPS (Spark Plasma Sintering), microwave sintering, hot pressing and laser sintering etc.

Although the laser sintering technique can be applied to many powder materials, the scientific and technical aspects of the production process and the effects of parameters such as sintering speed on the microstructures of the materials during production are not yet known. This production method is accompanied by multiple heat, mass and momentum transfers and chemical reactions that make the process very complex. When this situation is evaluated, this method used is basically based on experimental and experimental information. All these results show that the further development of this process is largely dependent on a better understanding of the concentration mechanisms and production parameters. There are different studies on this subject in the literature [4]. As mentioned earlier, another sintering method is Spark plasma sintering. SPS is a newly developed synthesis and process technique that makes it possible to sinter and sinter-bond at low temperatures and short times by charging the spaces between powder particles with electrical energy and the effective application of spark plasma is produced instantly at high temperatures. Like spontaneous high- temperature synthesis and microwave sintering, it enables the powder to be heated internally and by itself and is considered a fast-sintering method. SPS system has advantages such as ease of process, full control of energy and sintering temperature, high efficiency, reliability and safety compared to traditional sintering methods such as hot press and hot isostatic press [5]. The basis of the SPS process is based on the electrical spark discharge phenomenon. As a result of the high energy, the spark strike creates spark plasma at a certain temperature. Compared to hot press and hot isostatic press, SPS has shown significant improvements in intergranular bonding by collecting high energy pulses in one place. It allows sintering at lower temperatures between 200°C and 500°C compared to traditional sintering methods. Evaporation, melting and sintering is completed between 5- and 20-minutes including temperature rise time [6, 7].

The use of microwave for heating started with the heating of nutrients and then applied to many different materials such as polymers, ceramics and composites. After this development, it is used in the joining, sintering and coating processes of metallic materials. However, the use of microwaves in metallic materials is a challenging process due to the ability of metallic materials to reflect electromagnetic waves [8]. However, the use of microwaves has enormous potential for the discovery of unique microstructures and new materials. Microwave energy is a form of electromagnetic energy with a frequency ranging from 300MHz to 300GHz and a wavelength between 1mm and 1m. Microwave heating is the method by which materials interact with microwaves to absorb volumetric electromagnetic energy and convert it to heat. This is a different mechanism from traditional methods. In conventional heating, heat is realized by conduction, radiation and convection mechanisms between objects. In conventional heating, the surface of the material first heats up and continues as the heat moves into the material, that is, a temperature gradient occurs from the surface to the interior, but in microwave heating, the microwave heats the entire volume that penetrates the interior of the material. This heating mechanism provides efficient diffusion processes, low energy use, very fast heating regimes and relatively low process, lower sintering temperatures, improved physical and mechanical properties, convenience, homogeneous properties. It is preferred because of its advantages such as environmental damage. These advantages provide superiority to conventional methods [9]. Recently, material processing technology using microwave method has been the focus of attention due to its low production costs, energy and time savings, fast sintering time, high energy efficiency, product homogeneity and high efficiency compared to other production methods [10-12].

As stated before, different sintering techniques are encountered in the literature in the production of powder metal parts. In the researches, no studies on resistance welding technique were encountered in the production of metal powder parts under atmospheric conditions. Therefore, in this study, samples obtained from Fe powders by powder metallurgy method at various pressures were sintered using an electrical resistance welding machine in atmospheric environment. Thus, the sinterability, mechanical properties and microstructures of the sintered samples in new technique that was used first time in this study were examined.

2. MATERIAL METHOD

In this study, commercially pure and spherical iron powders with an average particle size of 60 μm were used. Iron powders are shaped in the mold by using a one-direct press at various pressures (650, 850, 1050 MPa) in the dimensions given (15x15x4 mm) earlier. The green densities of the samples after shaping were calculated using 0.0001 precision scales and caliper. The d= m/v formula was used for calculation of the green and sintered density. For this purpose the weight and volume of samples were determined after the pressing process. Then, the approach to theoretical density was evaluated according to this result. The samples were sintered at 1000 °C for approximately 100 seconds in an open atmosphere. During the sintering process, the temperature of the samples were measured using a thermocouple. During the process, the pressure on the electrode is applied by manually with the help of an external pedal but not observed pressure value. After sintering, they were cooled down to room temperature. In order to determine the sintered densities of samples, their weights and heights were noted and thus, the sintered densities were calculated. In addition, the Brinell hardness of the sintered samples was measured. For metallographic examination, the sintered samples were cut along the axis. After the grinding and polishing, sample was etched with 10% Nital solution. The resistance heating welding machine has a circular electrode. Therefore, the sintering behavior of the samples were examined with optical images taken from several region (non-contact and resistance contact region). Therefore microstructural characterization was carried out with the help of an Olympus optical microscope.

3. RESULT AND DISCUSSION

3.1. Evaluation of Density and Microstructure

Measured green and sintered densities of samples which was conducted to investigate the sinterability of samples pressed at different pressures by using a resistance spot-welding machine with electrical resistance heating technique, are given in Table 1. In addition, the relationship between pressing pressure and green and sintered density of samples are shown graphically in Figure 1.

Table 1. *Green and sintered density of samples sintered at various pressures*

Pressure (MPa)	Green Density (%)	Sintered Density (%)
650	88.39	91,17
850	92.27	96,11
1050	94,5	98,01

As can be seen in Table 1 and Figure 1, both green densities and sintered densities of samples increased with increasing pressing pressure. The average green density, which was determined as 88.39% at 650 MPa pressure, was found to be 92.27% with an increase of 3.88% at 850MPa pressure. When the pressure was increased to 1050MPa, the green density, which was determined as 94.5%, increased by 2.23% compared to that of the sample under 850 MPa pressure. Looking at sintered densities, for 650 MPa, sintered density increased by 2.78% to 91.17%, at 850 MP by 3.84% from 92.27% to 96.11%, at 1050 MPa by 3.51% from 94,5% to 98,01%.

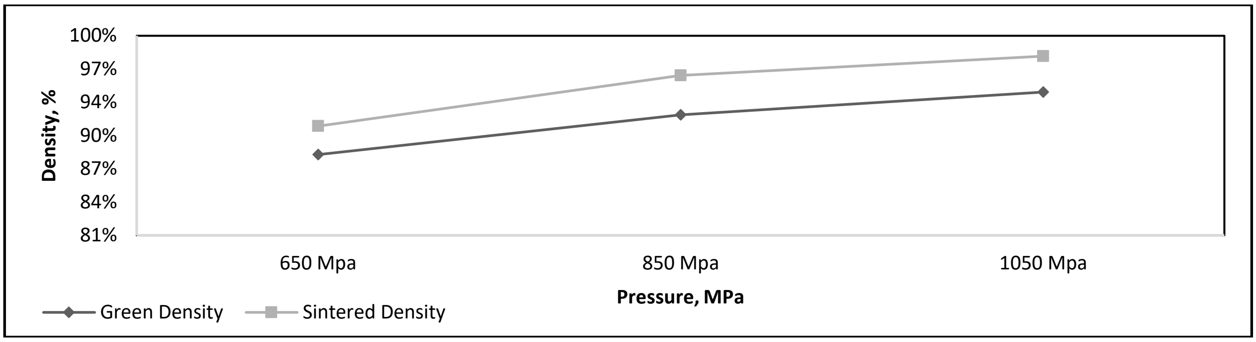
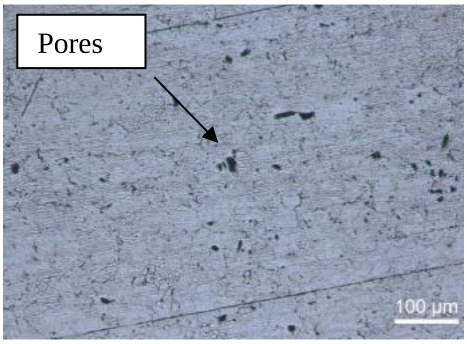


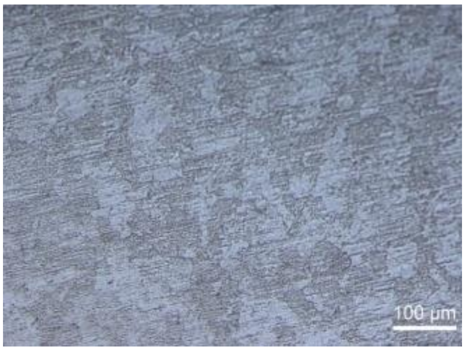
Figure 1. *Green and sintered densities of samples sintered with resistance heating technique*

In addition, the data indicate an increase of 6.11% in green density and 6.84% in sintered density from 650MPa, the lowest compression force used in the study, to 1050MPa, the highest compression force [13]. The fact that the rates of increase in green and sintered densities of samples are found close to each other. The findings indicate that the processes used in first time in this study are suitable. Although, there are difficulties in reaching densities above 95% sintered densities in powder metal samples; it can be regarded as the success of this technique in sintering that it reaches 98% densities in electrical resistance heating sintering technique. The reason for the high densities here can be explained by the fact that the electrically conductive electrode jaws behavior like a hot pressure sintering process during resistance heating. The microstructure of the samples was examined to determine whether the sintering was successful. The microstructures of the samples pressed and sintered at different pressures are given in Figure 2. As can be seen from the microstructures investigation carried out two different regions of samples at each pressing pressure. One of them is named as the main metal and the other is named as under the electrode contact region.

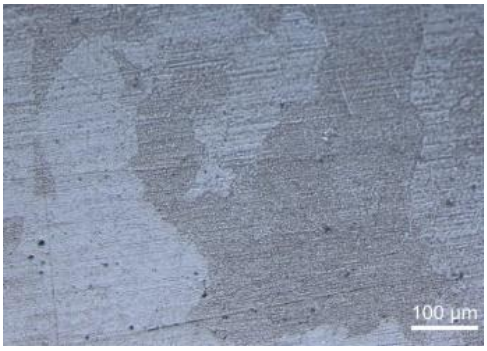


a) 650 MPa, x100 Non-contact region

b) 650 MPa, x100 Resistance contact region

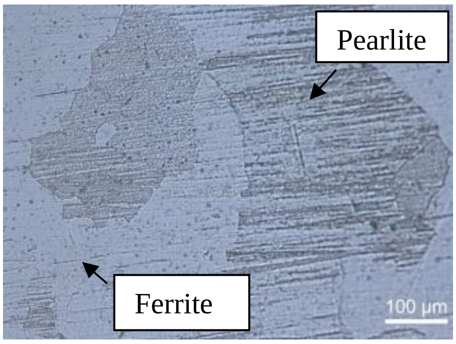


c)850 MPa, x100 Non-contact region



d)850 MPa, x100 Resistance contact region

e)1050 MPa, x100 Non-contact region



f)1050 MPa, x100 Resistance contact region

Figure 2*. Microstructures of sintered samples*

As seen Figure 2, the microstructures of sintered samples given for different pressure values have almost no porosity. As it can be understood from the microstructure images, there is no pore in the region under the electrode, while significant grain coarsening has occurred in the samples under high pressure. The reason for this is that it creates a driving force in grain coarsening during sintering, as the grain internal stresses are high at high pressures. In addition, because of the graphite powder used as a lubricant in the experimental stage, perlite phases were formed in the structure given in Figure 2.

3.2. Evaluation of Hardness

The hardness of sintered samples were measured from surface of the samples, on the electrodes contact area and main metal region. The data obtained are given in Table 2.

Table 2. *Hardness test results of sintered samples*

Pressure (MPa)	Hardness Value of Electrode Contact Region, (HB)	Hardness Value of Main Metal, (HB)
650	74	88
850	75	89
1050	76	90

As can be seen Table 2, the hardness determined from the electrode contact region, was measured approximately similar for each pressure value. In fact, the hardness was expected to be higher in samples pressed at high pressures [13], but it is understood that all samples showed approximately the same hardness value with sintering. It is found that hardness values measured from the electrode contact region is lower than base metal due to grain coarsening. The reason for this situation is temperature and the partial pressure applied to the electrode contact region during the sintering process. Because parameters such as density, micro hardness and grain size etc. depend on sintering temperature [14], heat input [15] and pressing pressure. In addition, the hardness increases in both regions as the pressure increases. When the studies in the literature are examined, it is seen that there is a relationship between hardness, microstructure and density. In this study, there is a relationship between the density values given in the previous section and the hardness results in this section. Because the density of the material and the amount of pores are inversely proportional. Therefore, it was determined that the mechanical properties of high density samples were better.

4. CONCLUSION

The following results were obtained in this study, which was conducted to investigate the sinterability of Fe powder with the electrical resistance heating technique;

1. It is found that powder metal samples can be sintered successfully in an unprotected environment with the electrical resistance heating technique.

2. Samples with high density values such as 98% can be obtained by new used sintering technique in an unprotected atmosphere.

3. It has been observed that the structures of the samples formed different grain size. Main metal region has got finer grain, while the contact regions under the resistance electrodes consisted of coarser grain structures. Moreover, the grain size of sintered samples increased by increasing pressure.

4. It is found that hardness values of the under the electrode contact region of sintered sample is lower than base metal region due to grain coarsening in associate with high heat input.

CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

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