Effect of Porosity, Density and Temperature on Microstructure and Mechanical Behavior of Hybrid Premix Sponge Ferrous Compact

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Abstract: This study on hybrid premix sponge ferrous sintered materials is focused on its mechanical properties and microstructure with respect to the amount of porosity. The pressure of 430 MPa and sintering temperatures 1090 °C, 1115 °C, and 1130 °C in a Nitrogen atmosphere for an hour is followed to produce this hybrid premix sponge ferrous compact. Radial crush strength, dimensional change and micro hardness test were performed on these samples to determine the mechanical properties. Scanning electron microscope (SEM) was used for fractured surface analysis, while light optical microscopy (LOM) is used to study the structure and pore formation. The study infers, as porosity percentage increased, tendency of irregular pore formation increased with simultaneous decrease in mechanical properties of the sample. Temperature of sintering has a vital role in decreasing the porosity of powder materials.

Keywords: Porosity, Density, Hardness, Microstructure, Sintering.

1. Introduction

Powder metallurgy is a net shape manufacturing approach, which is very attractive because of its ability to form powders directly into finished components. These components are formed in an economical manner having a considerable choice in material properties, mechanical, chemical, and microstructure control. The total porosity of the powder metal material depends mainly on compaction pressure, sintering temperature, sintering time, quality of metal powder, grain size, and its distribution [1-3]. Powder metal sintered components are preferred in industrial production due to their low energy consumption, minimum loss of raw powder, elimination of rejects, secondary operation, excellent size control and cost effectiveness. Among these elements ferrous powder is the most commonly used sintered component. Additionally, carbon with copper also improves the mechanical characteristics of the sintered components [4-7]. Powder metal components are usually

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produced by sintering process. However, a mixture of carbon and copper with ferrous powder greatly affects the sintered mechanical properties. Selecting and identifying the proper sintering process parameters is very important as it affects the quality of the product.

The main aim of the modern industry is to produce low cost, high quality products within a short time. The common method used for producing sintered parts is powder metallurgy and the sintering parameters are properly selected in order to produce any product with the desired quality. The density of the compact, the chemical composition of the materials, the sintering temperature, the sintering time as well as the use of lubricants are the parameters which will impact the sintering qualities like micro hardness, compressive strength of the product and microstructure [8]. Sintering temperature and carbon content played a major role in determining the mechanical and homogeneous microstructure [9]. Addition of 0.6 % Carbon yielded a tensile strength of 275-500 MPa and transverse rupture strength of 640-1260 MPa [10]. Addition of Brass 10 % with iron plays a major role in determining the mechanical properties and microstructure with an optimum sintering temperature less than or equal to 1000 °C [11]. Addition of Cu, C, and Sn with iron powder improves the mechanical and microstructure property. At higher temperature and graphite content, formation of cementite takes place [12]. Sintering temperature of 1140 °C with 3Mn-0.5Mo-0.7C obtains acceptable microstructures and good mechanical strength [13]. Addition of Ni and sintering temperature of 1250 °C with diffusion alloyed iron powder forms a Ni-rich phase and to overcome this problem addition of Cr eliminates soft Ni-rich areas. Tensile strength is improved from 626 to 801 MPa [14]. Addition of copper with iron powder plays a major role in swelling of the compact and carbon addition restricts growth of copper [15].

Very few works have been reported about the effect of porosity, density, sintering temperature on hybrid ferrous powder especially on mechanical and microstructure. The present work investigates the influence of porosity on the microstructure and properties of powder metal hybrid sponge ferrous powder samples. Mechanical behavior and microstructure were analyzed depending on the porosity amount. In addition, the pore morphology and fractured surfaces of the radial crush strength samples were investigated using SEM, and light optical microscope (LOM).

2. Materials and methods

The hybrid premix sponge ferrous powder used in this study has a chemical composition of Fe -NCI 100.24 – 75 % and SCI 100.26 – 25 % with 0.8 % Carbon, 2 % Copper and 0.8 % Zinc stearate as lubricant. A 50 KN capacity double action press was used to press the powder as per ASTM standard [16] and the average green density was found to be 6.6 g/cc as per ASTM standard B311-93 [17]. Similarly 1st group, 2nd group and 3rd group samples were produced from the compressed samples at a sintering temperature of 1090 °C, 1115 °C and 1130 °C respectively in a furnace with nitrogen gas atmosphere.

Cross head speed of 5 mm/min was used while measuring radial crush strength in the universal testing machine. Samples from all the three groups were used to obtain the results. Experiments were carried out as per ASTM standard B 331-95 at room temperature [18] and change in dimensions determined both green to sinter components. The recorded data are the average values of dimensional change for every set of three samples. Measurement of micro hardness values of these specimens were obtained by applying a load of HV1 for 15 sec duration as per ASTM standard [19] in a Vicker’s micro hardness tester (Matsuzawa MMTX7 B type, Japan).

Samples prepared according to standard metallographic procedures were investigated using (Leica DM 2500 M) metallurgical light optical microscope and their microstructures were analyzed. Fracture surfaces obtained from radial crush strength test were also analyzed by scanning electron microscope (SEM, JSM – 6610) for various magnification ratios.
3. Result and discussions

The density of hybrid premix sponge ferrous powder samples prepared by sintering in a nitrogen atmosphere at 1090 °C, 1115 °C, and 1130 °C for duration of 1 hour were determined using Archimedes principle. The average sintered density of the three samples were determined as 6.62 g/cc (92 % theoretical density), 6.65 g/cc (92.42 % theoretical density) and 6.7 g/cc (93.12 % theoretical density) respectively. Table 1 depicts the porosity and density values of hybrid premix sponge ferrous powder materials sintered at different temperatures. A similar study of Fe-Mo-Ni steel powder sintered at 1120 °C for 30 min showed 7.53 g/cc density [20]. Another study at 1140 °C and 40 min of sponge iron powders showed a density of 7.2 g/cc [21]. Fig.1 shows the impact of sintering temperature on density for the three different sample groups. It is noticed that the density increases proportionally with the increase in sintering temperature.

<table>
<thead>
<tr>
<th>Group</th>
<th>Sintering temperature, °C</th>
<th>Theoretical density, g/cm³</th>
<th>Sintered density, g/cm³</th>
<th>Porosity, %</th>
<th>Relative density, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st</td>
<td>1090</td>
<td>7.186</td>
<td>6.62</td>
<td>8</td>
<td>92</td>
</tr>
<tr>
<td>2nd</td>
<td>1115</td>
<td>7.186</td>
<td>6.65</td>
<td>7.58</td>
<td>92.42</td>
</tr>
<tr>
<td>3rd</td>
<td>1130</td>
<td>7.186</td>
<td>6.7</td>
<td>6.88</td>
<td>93.12</td>
</tr>
</tbody>
</table>

Fig. 1. Influence of Sintered temperature upon density of Hybrid Premix Sponge ferrous powder compact.

Optical micrographs of the hybrid premix sponge ferrous powder samples viewed under optical micrograph show the different pore distribution, porosity and powder grains for different group of samples sintered at 1090 °C, 1115 °C, and 1130 °C for a duration of 1 hour are shown in Fig. 2. Several variables namely green density, sintering temperature duration, alloying materials and particle size influence the nature of porosity [22-24]. Types of pore, pore shape and size, and the powder grain size have vital effect over the mechanical properties of powder metal components [25-27].

Samples sintered at 1090 °C, showed more irregular shaped pores with sharp edges. Large sized pores are found among powder grains marking indefinite grain boundaries. This microstructure leads to lower values of mechanical properties.
Fig. 2. Change of pore size in the microstructure of Hybrid Premix Sponge powder compacts at: (a) 1090 °C, (b) 1115 °C, and (c) 1130 °C for 1 hour.

This is due to improper sintering. Pores of irregular shape are also seen in samples sintered at 1115 °C. Non homogenous grain size distribution and indefinite grain boundaries are predominant. Thus both 1st and 2nd group of samples showed reduced micro hardness and radial crush strength resulted due to high porosity and irregular pore formation. On the other hand a spherical pore with low porosity is noticed in samples sintered at 1130 °C. This results in a remarkable enhancement of mechanical properties like micro hardness and radial crush strength. Fig. 3 depicts the sintering temperature effect on porosity for the samples of all the three groups. It can be concluded that samples sintered at higher sintering temperature have grain boundaries and pore geometry in acceptable terms.

Fig. 3. Influence of sintered temperature upon porosity of Hybrid Premix Sponge ferrous powder compact.

Fig. 4 shows the SEM micrograph of hybrid premix sponge ferrous powder samples. Structure of samples sintered at 1090 °C viewed under SEM micrograph reveals capillary and sharp edged pores. This is due to poor sintering and it has negative impact on mechanical properties of the sample group. On the other hand numerous sharp edged pores are noticed in the grain boundaries in the sample sintered at 1115 °C. This is due to inadequate sintering and it shows a decrease in material strength. Samples sintered at 1130 °C when viewed in SEM micrograph showed pores along the boundary of the grain with a reduction in pore size thereby more spherical in shape. This is due to increased sintering temperature and its effect on mechanical properties which is positive. Comparison of the 3rd group samples in SEM shows that samples sintered at higher temperature of 1130 °C have better mechanical properties.
The values of radial crush strength of the three different sample groups for varied porosity are given in Fig. 5. The measured value of radial crush strength of the 1\textsuperscript{st} group sample with density of 6.62 g/cc (8 \% porosity) was found to be 163.9 MPa. For the 2\textsuperscript{nd} group sample having density of 6.65 g/cc (7.58 \% porosity) was 174.59 MPa and that of 3\textsuperscript{rd} group sample with density of 6.7 g/cc (6.88 \% porosity) was 199.51 MPa. From the above values it is inferred that, increasing the density with relative decrease in porosity increases the radial crush strength. This is well agreed with earlier investigation [28-30].

Increase in porosity of the samples showed a decrease in strength values that decreases the ductility. The fracture surface analysis using SEM is performed to observe the porous structure of the samples obtained from compressive test. The result after the analysis is shown in Fig. 6. Increase in pore size and porosity \% during metallographic analysis resulted in a decrease in the strength of hybrid premix sponge ferrous materials. The sample of the 1\textsuperscript{st} group with 8 \% porosity, fracture surface analysis showed the least sinter neck formation and the greatest pore size. This is clearly visible in Fig. 6. The most ductile structure was noticed in the 3\textsuperscript{rd} sample group with 6.88 \% porosity in Fig. 6c.

The reason for ductility increase in the 3\textsuperscript{rd} sample group having 6.88 \% porosity is low porosity, regular spherical pores, large sinter neck formation and reduced pore size. Increasing the density with decrease in porosity can aid uniform pore formation along with pore size reduction as seen in Fig. 2 and Fig. 6. On the other hand irregular formation of pores with reduction in porosity resulted in reduced micro hardness and lower radial crush strength. Samples of 1\textsuperscript{st} group with 8 \% porosity showed the most irregular pores while samples of 3\textsuperscript{rd} group with 6.88 \% porosity had regular pore formation. Sinter neck fracture easily occurred in
the 1st group sample because of the reduced strength and micro hardness due to irregular pore formation. This trend is in agreement with the work of others [31-33].

![Fracture surface of radial crush strength samples](image)

**Fig. 6.** Fracture surface of radial crush strength samples at: (a) 1090 °C, (b) 1115 °C, and (c) 1130 °C for 1 hour.

Similar studies reveal that irregular pore structure leads to reduction in strength and ductility with an increase in porosity of powder metallurgy parts [34]. Bekir Yalcin states that the criticality of pores in components used in industries are risky since they have poor mechanical properties. Therefore, it is necessary that pore must be controlled. According to the study of Bekir Yalcin, increased porosity resulted in decrease of all mechanical properties [35]. Further studies report that determination of sintered parts porosity used in industrial applications is vital [36]. Naci Kurgan [37] suggests that due weight age should be given to the porosity of 316L stainless steel implant samples to evaluate desired microstructure and mechanical properties. They conclude that a porosity of 12.29 % in P/M 316L stainless steel materials resulted in enhanced mechanical properties making it suitable for implantation. In a similar manner this study hybrid premix sponge ferrous powder reveals that 6.88 % porosity (6.7 g/cc) has the best mechanical properties in comparison with other samples.

![Influence of sintered temperature on dimensional change](image)

**Fig. 7.** Influence of sintered temperature on dimensional change of Hybrid Premix Sponge ferrous powder compact.

Fig. 7 shows an increase of 0.103 % from green to sinter size in the linear measurements of test specimens sintered at 1090 °C due to diffusion of carbon in the iron matrix. The presence of ferrite and pearlite structure confirms the same in the photomicrograph of the test specimens at 1090 °C in Fig. 8a. Near the pores, presence of
unmelted copper was noticed. When the sintering temperature increased to 1115 °C, it showed an increase in linear growth of about 0.113 % which was caused because of the melting and diffusion of melted copper in the iron-carbon alloy as shown in Fig. 8b and the photomicrograph confirms that unmelted copper is absent in the structure, which shows that there has been a complete diffusion of carbon in iron matrix and is proved by the absence of ferrite in the structure and a subsequent particle to particle bonding was seen. When the temperature increased to 1130 °C, the growth increased by 0.118 % which is shown in Fig. 8c and it is due to near complete particle to particle bonding or sintering. The fact is that a temperature of 1130 °C is needed to obtain complete sintered structure. The observed values are in compliance with the values reported in the earlier work [28, 32, 33, 38-41].

**Fig. 8.** Microstructure of sintered Hybrid Premix Sponge powder compact at: (a) 1090 °C, (b) 1115 °C, and (c) 1130 °C for 1 hour.

Fig. 9 shows the micro hardness of the sample groups. Using the first and second group of sintered samples with 6.62 g/cm³ density with 8 % porosity and 6.65 g/cm³ density with 7.58 % porosity were used to obtain the average micro hardness values of 127HV and 136HV. The third sample with sintered density of 6.5 g/cm³ and 6.88 % porosity showed an average micro hardness of 140 HV. It was noted that the micro hardness increased according to sintering temperature. The surface of the samples on which the micro hardness values were measured was related to the density due to compacting for P/M samples, powder lubrication condition and mechanical properties of the powder [32, 42-46]. It can be concluded that the micro harness was directly related to density and inversely related to porosity.

**Fig. 9.** Influence of sintered temperature upon Micro hardness of Hybrid Premix Sponge ferrous powder compact at 1090 °C, 1115 °C, and 1130 °C for 1 hour.
4. Conclusion

Systematic study of the impact of porosity on the mechanical behavior and microstructure of hybrid premix sponge ferrous powder was carried out successfully. From the results obtained for the three different sample groups with varying porosity, it can be concluded as follows:

1. Increase in radial crush strength and micro hardness occurs when sintered density increases. Reduced pore size and low pore clustering is the reason for the increase in radial crush strength, since they act as stress concentration centers.
2. In low density samples premature failure occur due to strain localization because of large irregular highly clustered pores. More homogeneous distribution and spherical pores result in uniform plastic strain distribution in samples with high density.
3. Lower pore fraction, reduced average pore size and uniform spherical pore shape resulted with increase in sintered density. Increase in size of pore is directly co related to the increase in pore shape irregularity. Finally, based on the results and analysis the best mechanical properties like radial crush strength and micro hardness can be achieved in powder metal component with porosity 6.88 % when compared to other samples (8 % and 7.58 % porosity).
4. The important parameters that influence the detection of porosity of powder materials are sintering temperature and compacting pressure. Finally decreasing the porosity of sintered powder metal material shows the improvement in the microstructure and mechanical properties of the components.

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5. References


Садржај: Студија о хибридном феро синтерованом материјалу је фокусирана на механичким својствима и микроструктури у односу на количину порозности. Притисак од 430 MPa и температуре синтеровања 1090 ºC, 1115 ºC и 1130 ºC у атмосфери азота током једног сата, је процедура добијања синтерованог узорка. Чврстоћа, промене димензија узорка и микрочврстоћа су рађени на овим узорцима да би се одредила механичка својства. Скенирајућа електронска микроскопија је рађена на прелому, док је оптичка микроскопија употребљена ради проценирања микроструктуре и формирања пора. Док проценат порозности расте, повећано је формирање нерегулярних пора неправилног облика, са симултаном смањењем механичких својстава узорака. Температура синтеровања има кручнију улогу у смањењу порозности материјала.

Кључне речи: порозност, густина, чврстоћа, микроструктура, синтеровање.

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