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Evaluation of the characteristics of Iron (Fe) Metal Powder injected parts using HDPE and Thermoplastic Rubber (TPR)

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ABSTRACT
Metal Injection Molding (MIM) is a process of powder metallurgy, similar to the plastic injection process. This process is ideal for obtaining parts of complex geometry, difficult to machinability, small batches with finishing and final dimensions, or not. The MPI, part generation process, has several steps that must be carefully followed to avoid errors that compromise product quality. The objective of this work was to produce parts with different polymers in a binder, High-Density Polyethylene (HDPE), and Thermoplastic Rubber (TPR) and after comparing them. Results obtained in this research showed that the parts injected with HDPE showed no defects after injection. The parts injected with Thermoplastic Rubber offered, according to the obtained results, an excellent mechanical resistance when compared to those that used HDPE.
Keywords – Metallic Powder, Powder injection, Polymers, Granulometry.

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I. INTRODUCTION

According to many companies, seeking an increase in their profits, are seeking greater assertiveness in product quality and speed of delivery by investing in smart factories [1]. In these factories, technology and innovation are part of everyday life, offering the manufacture of various types of products in less time. This idea reinforces the studies where the author studied the equipment utilization planning rate, where, according to the same author, the assembly of a manufacturing plant depends on the level of aggregate demand [2].

Competitiveness in industries refers to the need to seek innovations faster and more efficiently. The term innovation that is originated from the Latin "*innovatione*" presents as a meaning renewal [3]. However, it is believed that innovation represents the application of creativity; that is, first comes creativity and then innovation [4].

In the area of manufacturing, in particular, it is plausible that engineers will develop new materials and processes that will contribute to corporate profitability in line with sustainable resource consumption. The Metal Injection Molding (MIM) process is one of the subdivisions of powder metallurgy and is a process similar to the polymer injection process. Historians show that such an operation had been carried out in the manufacture of spark plugs in the 1920s, but only in 1979 was their industrial application and was used for large-scale manufacturing of parts by industry [5].

Metal injection molding, in view, combines the most useful features of powder metallurgy and plastic injection molding to facilitate the production of complex small metal components while offering excellent mechanical properties [6].

Ratifying the advantages of the MIM process described earlier, it also mentions that the process offers several advantages over other manufacturing processes, such as design flexibility with minimal finishing operations and reduced material waste [7].

Research in this area is necessary, and its results are of interest to industrial-technological development. Thus, the study presented has as its premise to analyze the properties generated by the parts molded and injected by the MIM process under different mixing conditions where the iron base will have mixtures with HDPE (High-Density Polyethylene) and Thermoplastic Rubber.

II. MATERIALS AND METHODS

The present work had as main objective to study the obtained properties (hardness, microhardness, and density) in the samples produced from the injection of Iron Powder mixed with organic compounds. The compounds to which they belong are mainly HDPE and Thermoplastic Rubber (TPR) as an alternative binder. After the generation of the specimens, they were subjected to analysis of the mechanical behavior of the sintered material to compare the properties between the parts generated with HDPE and thermoplastic rubber that is the object of interest of this research. All specimens were mixed with iron powder, having as main matrix the binder with HDPE and as an alternative the Thermoplastic Rubber (TPR).

The other polymeric components of the two binders remained the same for both matrices, namely Carnauba Wax, Paraffin, and Stearic Acid. Thus, we sought to evaluate whether thermoplastic rubber is a technologically viable alternative in the area of MIM for HDPE. To make the mixtures, we used the helical mixers (Haake) and the planetarium. Sample densities were measured on the densimeter.

Chemical extraction was performed in the greenhouse with the additional use of hexane. The thermal removal was done in a vacuum oven and sintering in an argon gas atmosphere furnace. The analysis of Brinell and Vicker's hardness properties was verified using specific durometers for each application.

1) Powder Injection Molding

The Powder Injection Molding (PIM) process is a derivative process, derived from the plastic injection process plus powder metallurgy [8]. In PIM, the feedstock is a mixture of a binder with metal or ceramic powders, and such a method allows the unique opportunity to manufacture high-quality ceramic components using plastic forming technologies [9]. The binder system is composed of post-inorganic and polymers and has the function of forming the vehicle for filling the mold. At the end of the process, the vehicle must be removed by chemical and thermal processes before sintering.

Some advantages of this process concerning other manufacturing processes [10]:
- Manufacture of parts with highly complex geometries;
- Low material loss;
- Wide range of materials available;
- Higher productivity.

Figure 1 illustrates in a flowchart the PIM process, from component mixing to part sintering.

Fig. 1: PIM process flowchart [11].

2) Powder Injection Molding (PIM)

Powder metallurgy can overcome casting and processing limitations and can produce a more exceptional controlled grain size [12]. The main processes of production of powders and describes in their perception, the most used as being: grinding, chemical methods, and atomization [13]. To understand the influence of the form of obtaining of the powders, some aspects appear in table 1.

Table 1: Production techniques of metal powders related to particle size, morphology and costs [14].

Technique	Average Particle Size (μm)	Morpholo gy	Cost
Gas Atomization	20 - 40	Spherical	High
Water Atomization	10 - 40	Rounded	Moderat e
Centrifugal Atomization	25 - 40	Spherical	High
Carbonyl	1-10	Spherical	Moderat e
Oxide Reduction	1-10	Polygonal	Moderat e
Precipitation	0.01 - 3	Polygonal	Low
Milling	0.1 - 40	Irregular	Moderat e

3) Binding system

Binder is a mixture of polymers and one of the significant factors in the processing flow of parts via conventional powder metallurgy, injection, or laser fusion [15]. The binder can be classified as short-chain (waxes and paraffin), characterizing the plasticity of the injection mass when injecting metallic powder and long-chain (polypropylene and polystyrene), which gives mechanical strength to the mixture [16].

In the binder removal step, the combination is chemically and thermally removed, and there should be minimal interactions (necks) between the dust particles before their removal, thus avoiding possible distortion or deformation of the part [17]. Figure 2 (a) and (b) show how the interactions (necks) between material particles are formed during feedstock homogenization.

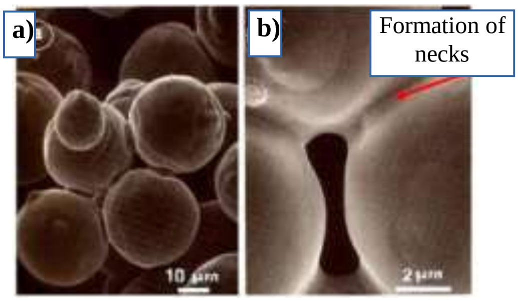

Fig. 2: Interaction between particles (a) and (b) [16].

4) Mixing Step

This first step consists of the homogenization of the components forming a dough (feedstock), and in this work, the Haake mixer was used. Interestingly, the relationship between metal dust and binder is intense, as this relationship will result in the properties of the feedstock [18]. The powder and binder are hot mixed above the melting point of the constituents, forming a homogeneous mass with a constant shear rate and reasonable interaction forces to provide a uniform coating on the powder surface [19].

The proportion of each material is essential because it defines how the feedstock will behave at the end of the process [20], corroborating the importance of the effect of the mixture on powder metallurgy, which has a significant impact on the enthalpy of the generated compound. The mixing time, as well as the shape of the mixer, are also influencing factors in powder mixing for powder metallurgy compound generation [21]. In metallic powder injection (PIM), a low amount of binder will generate a highly viscous material and, consequently, injection problems. However, if there is an excess binder, it will cause problems in the extraction process, such as high standard time and gaps, directly influencing the material flow and its final properties [22].

5) Injection Steps

This is the second step of the MPI process and consists of inserting the feedstock into the mold. The material is heated so that it has sufficient fluidity to be transported through a threaded screw, similar to the plastic injection process [23].

In the mold cavity, the injected load already takes the final shape of the part and is then cooled [24]. To avoid some particular problems of this process, some machine parameters must be taken care of and controlled: injection temperature, injection speed, mold temperature, molding pressure, molding time, mold cooling time.

6) Binders Extraction Step

At this stage, the vehicle or binder that is attached to the part will be removed, and the part will suffer a loss of strength, requiring delicate handling to avoid compromising the integrity of the sample [25].

The binder removal process represents a problematic step for all types of mixtures [26]. One of the reasons for adding different components in MPI is that it helps in the strength and fluidity during metal injection and helps in binder extraction. There are three commonly used methods for binder removal: solvent dissolution, temperature action, and catalytic action. The fastest way is by dissolving with organic solvent or water [27].

7) Sintering Steps

Sintering Steps is the last stage of MPI, and the bonding between particles should occur through densification, where mechanical properties will be attributed to the parts by chemical bonds [28]. The sintering step can be divided into three stages: In the first one, the neck's, interactions between the particles, there is little retraction of the piece. In the second moment, the particles start the grain growth, and the densification of the part occurs, in this stage, the pore shape is more rounded, and the appearance of isolated pores characterizes the beginning of the final stage [29].

Figure 3 illustrates how the material behaves and how the interaction (grain growth) occurs during the sintering phase.

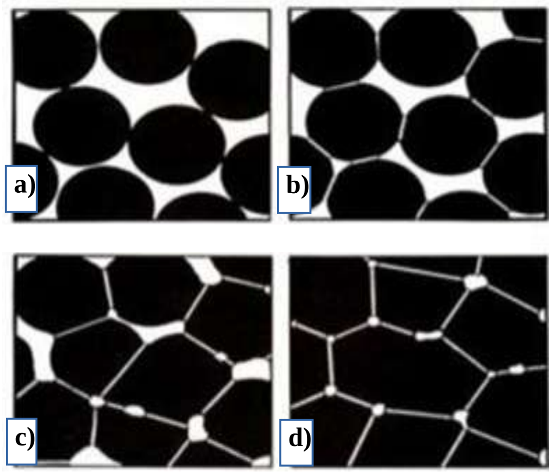

Fig. 3: a) Particles at the beginning of sintering. b) The first moment of sintering and grain growth. c) Shows how the particles behave during grain growth. d) End of sintering process and material grain growth [29].

8) Comparison of Polymers

Polyethylene is a partially crystalline, flexible polymer whose properties are markedly influenced by the relative amount of the amorphous and crystalline phases [30]. HDPE is a hard plastic and has high strength, melting temperature around 110ºC, and is an excellent alternative to work with injection or blow. The Thermoplastic Rubber has a good working range in temperatures between -60ºC and 135ºC without cracking or greasiness, it has higher impact absorption power, generating less noise in some parts. Both materials combine ecological and economic aspects [31].

III. METHOD USED FOR RESEARCH

All of the raw material was selected and checked so that there was sufficient quantity for binder weighing and forming. Subsequently, it began by considering the samples in separate pots for a total of 10 150g cups each to produce 1.5 kg of sample for each material (HDPE and Thermoplastic Rubber). For the generation of the specimens, two suppliers of a base material (iron powder) were used, and they classified as ('X') and ('Y'), respectively. After weighing the raw material of the components, the mixture was sent to the Haake mixer, where a homogeneous mass was formed and then sent to the crusher, and finally, the samples injected. With the samples ready, their densities were verified, and the chemical extraction started and later thermal extraction to then sinter the pieces. After the sintering process is completed, the specimens are ready for testing, which is the main objective of this research.

1) Powder Injection Molding

We selected the raw materials for weighing and then weighed the components in proportion according to Table 2.

Table 2: Proportion of MPs that used.

MP	Volume Ratio (%)	Weight ratio (%)
Iron Powder	63.18	84.2
Paraffin	19.93	7.0
HDPE or TPR	9.52	4.2
Stearic acid	5.79	2.5
Carnauba wax	4.57	2.1

After weighing the powder mixture plus the binder, the samples were mixed and homogenized in the Haake mixer.

2) Pelletizing the groundmass

After placing the feedstock in Haake to mix and homogenize, the mixer equipment delivers as a product a dense, homogeneous dough of varying sizes and some large, so it is necessary to pass the mixture into the knife pelletizer. The Mill supplied as a product the ready-to-inject filler, pelletized in small particles between 1 mm and 5 mm, non-uniform. This grinding is presented in Figure 4.

Fig. 4: Ground material.

3) Load Injection

The injector used is large equipment of HIMACO brand with high injection capacity, and the mold used was a swab specimen. The samples were for injection after being pelleted in the mill. In the injection, it was decided to use a temperature gradient in the injection gun (170 ºC - 180ºC -190ºC). The injection provided as products 4 (four) samples being 2 (two) thinner types, but with the same proportion, due to the mold. Some samples were taken to the densimeter to obtain the density after injection, before chemical extraction and then proceeded with the chemical extraction using hexane as a solvent and a parameter of 60 ºC with the samples submerged in the greenhouse for 4h (four hours). The densities of the samples were analyzed and compared.

In the chemical extraction, only the less dense and soluble components to the reagent were removed, that is, paraffin and carnauba wax, mainly. After chemical extraction, the samples are ready for thermal extraction and removal, mainly of HDPE and Thermoplastic Rubber. In this step, some residual wax or paraffin was also extracted. A temperature range was used, ranging from 525 ºC to 550ºC, starting at 25 ºC. The rate of change in temperature rise was 0.1 ºC per minute, totaling at the end of the thermal extraction process a total of 87.5h or 3.6 days for the extraction process to be completed.

In the thermal extraction process, the samples were subjected to a vacuum atmosphere, avoiding possible oxidation of the parts during the process, and their cooling occurred at room temperature.

4) Sintering

After thermal extraction, the samples were sintered to eliminate the pores in pieces.

In this process, the final temperature of 1200 ºC was used, remaining at this temperature for 1h with a heating rate of 10ºC per minute. The atmosphere was controlled using Argon gas. Its cooling occurred in a similar way to thermal extraction, i.e., inside the oven to room temperature.

5) Hardness Test

Two types of hardness tests were performed on the samples. The first was Brinell Hardness and the other was Vickers Hardness.

IV. RESULTS AND DISCUSSION

Four sample types were injected with two different powder types, two with HDPE in the binder and two with thermoplastic rubber. After the injection of the loads, the pieces were weighed, and their densities, weights, and volumes compared.

The injection mold shows where the injections of the pieces of this research were performed 4 (four) cavities, 2 (two) smaller, and 2 (two) larger.

The graph in figure 4 shows the volume, weight, and density of the samples immediately after injection. The samples were classified as S_1 (large HDPE), S_2 (small HDPE), S_3 (large thermoplastic rubber), and S_4 (small thermoplastic rubber).

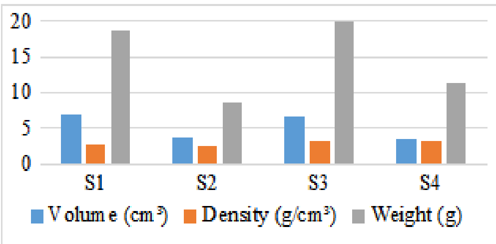

Fig. 4: Graph of volume, weight, and density after injection.

Graph 2, on the other hand, shows the same variables, but now, after chemical extraction.

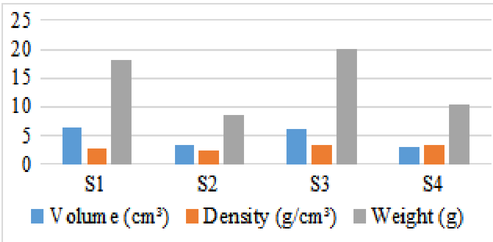

Fig. 5: Graph of volume, weight, and density after chemical extraction.

With this, we arrived at the graph of figure 6, which shows the percentage of material removed, that is, the variation of volume, density, and weight in chemical extraction.

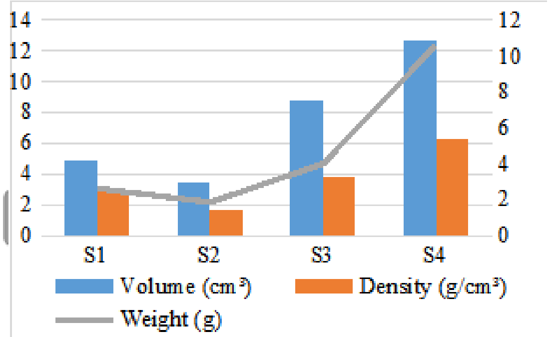

Fig. 6: Graph of chemical extraction variation in percentage.

The generated specimens were submitted to Brinell hardness and Vickers microhardness test and analyzed under the microscope where the following data were collected:

• Brinell Hardness: obtained an average of 88 Rockwell Brinell in the samples with the 'X' powder. Already the samples that used the raw material (powder) of the company 'Y', it was not possible to perform the test;

• Vickers Microhardness: An average of 62 Vickers to 181 Vickers was found, with the HDPE and thermoplastic rubber samples using the company's raw material ('X').

The other samples, where the raw material used was from the company ('Y'), did not allow analysis because it offers excess pores and gaps in the generated samples.

After identification with the durometer, the necessary calculation was performed to identify the resistance of the material. For the capture of the identified area, a microscope was used where the samples were analyzed and then obtained the necessary diameters to perform the calculations and obtain the value of the Vickers microhardness. Equation 1, shown below, was the equation used for such a situation.

$$HV\ 0.1 = 1.8544xP/d^2 \qquad (eq.1)$$

- HV 0.1: identifies Vickers hardness with 100 kgf applied load;
- P: represents the weight force applied in (kg) by the identifier on the part;
- d: are the average between the two diagonals in (mm).

Thus, we arrived at the following results presented in the graph of figure 7.

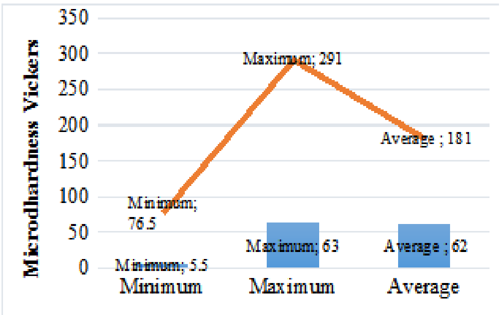

Fig. 7: Graph of the average Vickers microhardness obtained for the powder samples ('Y').

V. CONCLUSION

It can be observed in this research that the injection of iron powder with HDPE and thermoplastic rubber as binders presented satisfactory behavior, although there are defects in some specimens. HDPE samples showed no injection defects; such a result was expected because the material is usually in the industry and its known behavior. The specimens with thermoplastic rubber showed good mechanical strength in hardness tests after sintering, showing stability during thermal extraction. Thus, the research showed based on the proposed tests, and the results obtained that thermoplastic rubber can replace HDPE in the injection process of metal powders. Thereby, thermoplastic rubber may replace HDPE in the current commercial process and may generate, with their studies for this purpose, an expectation of a lower production cost.

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