Metal injection moulding of 17-4PH stainless steel: Effects of porosity on the mechanical properties of the sintered products

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Abstract. 17-4PH stainless steel (SS) is a martensitic precipitation hardenable alloy which normally contains approximately 15.5-18.5 wt.% Cr and 2.5-4.5 wt.% Ni and Cu. It is well known for its high impact strength and fracture toughness, and excellent corrosion resistance at service temperatures below 300 °C promoted by its Cu-rich precipitates. 17-4PH SS is widely used as a structural material in various sectors such as aerospace, medical, petrochemical, etc. The high strength of the alloy makes it difficult to machine, especially when making complex shaped products. Metal injection moulding (MIM) uses the shaping advantage to produce small-to-medium intricate products in high volumes at a relatively low cost. However, due to the nature of MIM process, the mechanical properties of the sintered products depend strongly on various metallurgical MIM variables with porosity included. Porosity is largely dependent on the starting powder characteristics, powder loading, and sintering conditions. In this study, the mechanical properties of the sintered 17-4PH SS MIM samples fabricated at different powder loadings, viz. 55, 60 and 65 vol.%, will be evaluated. The starting powder particle size distribution is -5, -15 and -45 µm. The sintered density, microstructure and hardness response of the samples are presented. These properties are influenced by the nature of the pores, i.e. size, density and distribution.

1. Introduction

17-4PH stainless steel (SS) is a precipitation hardenable martensitic alloy widely used in areas requiring high strength, excellent corrosion resistance and good biocompatibility such as automotive, aerospace, chemical and power plants, and medical industries [1], [2]. The alloy typically contains 15.5 – 18.5 wt.% Cr and 2.5 – 4.5 wt.% Ni and Cu elements which give the alloy its unique properties [3]. This alloy is, however, difficult to machine especially when making intricately shaped products. Metal injection moulding (MIM) uses the shape advantage to produce small-to-medium complex metal parts at a relatively low cost in high volumes. MIM combines powder metallurgy (P/M) and plastic injection processes to fabricate these metal products [4]. Four main stages in MIM include mixing, injection moulding, debinding and sintering. The mechanical properties of the sintered products strongly depend on the porosity, inclusions, oxygen content and other metallurgical variables [5]. By varying the sintering conditions, MIM process can allow injection moulded parts to have a wide range of porosity level which can provide a very useful scope to explore and investigate the mechanical properties of the sintered products. Furthermore, the starting powder loading and particle size were also investigated on
how they affect the porosity level and final mechanical properties. Sung et al. [2] studied the influence of sintering temperature on the porosity level of 17-4PH SS and discovered that sintering temperature influences the shape and distribution of the pores. Mukund et al. [6] investigated the effect of bimodal powder particle size distribution on the rheological properties of the feedstocks, and defects and distortion evolution during sintering. Other studies examined the powder size variation at fixed powder loading against the rheological properties [7] and final mechanical properties [8].

Porosity remains a critical topic for MIM practitioners and end-users of MIM products. This work examines a combination of powder loading and powder particle size on the porosity level and hardness response of the final sintered parts.

2. Materials and experimental methodology

Precipitation hardenable martensitic stainless steel (17-4PH SS grade) powders of different sizes viz. -45 µm (supplied by Praxiar Surface Technologies), -15 µm and -5 µm (supplied by Epson Atmix Corporation) were used as starting materials in this study. The typical chemical composition of the 17-4PH SS powders is given in table 1. The powders were analyse for size and morphology using a scanning electron microscope (SEM, Jeol). For feedstock preparation, the powder materials were homogenously mixed with a polymeric binder at a ratio of 55, 60 and 65 vol.% with the binder reducing, and the feedstocks were labelled 55a, 55b, 55c, 60a, 60b, 60c, 65a, 65b and 65c; whereby a, b and c resemble the powder particle size -45, -15 and -5 µm respectively. The feedstocks were injection moulded into a ‘dog-bone’ shape.

Table 1: Typical chemical composition of the starting 17-4PH stainless steel powders

| Elements (wt.%) | C | Si | Mn | Ni | Cr | Cu | Nb | P | S | Fe |
|----------------|---|----|----|----|----|----|----|---|---|----|
| 17-4PH SS (Praxiar) | - | 1.0 | - | 4.0 | 16.0 | 3.0 | 0.25 | - | - | Bal. |
| 17-4PH SS (Epson) | <0.07 | <0.5 | <0.3 | 3.0-5.0 | 15.5-17.5 | 3.0-5.0 | 0.15-0.45 | <0.04 | <0.03 | Bal. |

The injection moulded green parts were chemically and thermally debound to remove the polymeric binder components prior to sintering. The samples were pre-sintered at 1000 °C for 2 hours to remove the residual binder. Sintering was done under a vacuum furnace at 1300 °C for 3 hours. Sintered samples were cut-off for metallographic analysis, density and hardness evaluation. Sintered density was done through Archimedes’ principle using OHAUS explorer precision electronic balance. Polished samples were observed under optical microscope for porosity analysis, and the samples were further taken for micro-Vickers hardness testing at a load of 0.5 kgf.

3. Results and discussion

The as-received 17-4PH stainless steel powders were analysed for size and morphology, as depicted in figure 1. All the powders were spherical, however, the finer powders viz. figure 1b and 1c had agglomerated and this could be attributed to their larger surface area to volume ratio which is proportional to the strength of the cohesive forces [9]. Agglomeration leads to poor packing and flowability of the powder particles [7]. During injection moulding, higher shear rates between 10² –
10^3 s\(^{-1}\) [6] are introduced to break-off the agglomerates and improve homogeneity and feedstock stability [7], [10]. It is expected that finer particle size will improve sinterability and hence reduce porosity in the final sintered products.

Figure 1: SEM images showing morphologies of the as-received 17-4PH stainless steel powders

Figure 2 gives the optical micrographs of the as-sintered parts. It can be seen that porosity in MIM products is inevitable. The size of the pores reduces with particle size (see 55a to 55c, 60a to 60c, etc. for example). However, there is no significant change in terms of porosity size between feedstock 55a and 55b. A decrease in pore size from lower powder loading to higher loading is attributed to lesser binder content leading to closely packed arrangement of the particles and more contact surface areas between the powder particles. In addition, closely packed arrangement of the particles (i.e. less binder content) and finer powder size contributed to finer microstructure and pore size (55c, 60c and 65c), and this result gives a good indication of better mechanical properties.
Figure 2: Optical micrographs of the as-sintered specimens

Figure 3 show the density variation of the as-sintered products. Generally, feedstocks -45 µm gave the least sintered density. The -15 µm feedstocks give good densification with 60b and 65b possessing the highest sintered densities. It can be deduced that densification improves with smaller particle size. The improved sintered density is attributed to better sinterability of the finer powder particles.

The porosity level of the as-sintered products can be estimated by using the following equation [11]:

\[
Porosity = \left( 1 - \frac{\rho_S}{\rho_H} \right) \times 100\%
\]  

(1)

Where \( \rho_S \) is the apparent density of the as-sintered samples and \( \rho_H \) is the apparent bulk or pycnometer density. The pycnometer density (\( \rho_H \)) of 17-4PH SS is 7.75 g/cm\(^3\). Using equation (1), the lowest porosity levels recorded is 2.97% and 2.94% for 60b and 65b respectively. Feedstocks 55a and 65a had the most porosity levels at 5.74% and 5.63% respectively.

Figure 3: Density profile of the as-sintered parts
In Figure 4, the hardness profile of the as-sintered feedstocks is presented. The hardness improves with decreasing particle size from all the feedstocks. Powder loading does not influence, linearly, the hardness variation in the feedstocks. The 60c feedstock, viz. 60 vol.% -5 µm, has the highest hardness. Porosity does not have a significant effect on the hardness of the material.

![Figure 4: Hardness data of the different feedstocks after sintering](image)

Following the presentation and discussion of results, we can make the following general remarks:

- Porosity is important in some areas, particularly medical sector when building orthopaedic implants for good osseenetration [12]. Therefore, for high porosity level and size, larger powder particle size and lower powder loading (55a and 55b, figure 2) is important.

- For better mechanical properties such as increased hardness, higher powder loading and finer particle size is recommended (55c, 60c and 65c, figure 4). This is critical for applications where high strength is required such as automotive and aerospace industries.

4. Conclusions

From the work presented, the following conclusions can be drawn:

- Finer metal powders tend to agglomerate due to large contact surface area which promote strong cohesive forces between the particles.

- Higher powder loading contributed to the finer pore size after sintering and this was attributed to lesser binder content in the feedstock matrix which led to more contact surfaces between the powder particles.
• Finer powder particle size contributed to finer microstructure and pore size whereby feedstocks 55c, 60c and 65c gave the finest porosities. This effect resulted from better sinterability of fine powders.

• Porosity range obtained from the feedstocks lied between 2.94% - 5.74% whereby feedstock 65b gave the least porosity level and feedstock 55a gave the most porosity level.

• Powder loading influences the size of the pores only, whereas powder particle size significantly influences pore size and pore density, sintered density, and also hardness. Thus for high strength and hardness fine powder is recommended, and for porosity size and control (medical applications) powder loading variation is recommended. The hardness response of the material is not influenced significantly by the porosity level and size.

• Future work will consider the effect of porosity on tensile and fatigue behaviour as literature states that pores are often crack initiation sites and reduce fatigue life. Tensile and cyclic fatigue properties are known to be monotonic, hence reduction in fatigue life influences tensile strength. In addition, analysis of these pores using CT scans will also be considered.

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