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Metal injection moulding of a 17-4 PH stainless steel: a comparative study of mechanical properties

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Abstract. While the metal injection moulding (MIM) of the precipitation-hardenable 17-4 PH stainless steel has found wide-ranging applications in a number of industries and popularity as a workhorse alloy, data on the as-sintered properties 'that can be expected' is limited and scattered in the literature. A quantitative comparison of the mechanical properties of assintered 17-4 PH materials prepared via MIM is currently not available in the literature. -5 µm, -15 μ m, and -45 μ m starting 17-4 PH stainless steel powder materials and their bimodal mixtures were used to formulate with feedstocks investigated in this work. This paper compares the as-sintered mechanical properties resulting from the measured tensile tests against those properties reported in the literature, those specified in the ASTM and MPIF standard minimum specification for MIM materials, those properties specified in the material suppliers' datasheets and the mechanical properties of 17-4 PH wrought material - for completeness. While the as-sintered properties of the CSIR 17-4 PH material are generally lower than expected, the comparison shows that the measured properties are within the industry standard specifications. Findings from this study are a testament that a range of mechanical properties is feasible from the 17-4 PH alloy.

1. Introduction

The precipitation-hardenable 17-4 PH stainless steel has a remarkable combination of superior properties. It is the most common and most used type of the precipitation-hardenable stainless steels [1] and it suitably finds wide-ranging applications in a number of industries [2,3].

Many researchers have reported attempts to fabricate 17-4 PH components via numerous techniques alike. The challenges associated with fabricating of complex, near-net shaped 17-4 PH stainless steel components via the conventional press and sinter (P&S) and additive manufacturing (AM) techniques are well documented. These challenges include low powder compressibility for P&S [4] and the undue residual stress accumulation from martensitic transformation during a deposition for AM [1,5,6]. More recent reports have however widely reported on the attractiveness of metal injection moulding (MIM) in the fabrication of complex, near-net shaped 17-4 PH components, for example, Wu et al have reported MIM 17-4 PH stainless steel as a promising material for the industry because of its superior mechanical and corrosion performances [2]. The MIM technique is suitable for the costeffective manufacturing of high volume production of intricately shaped parts where high dimensional accuracy is required – further details are discussed elsewhere, see Ref.s [7–11].

The current article builds on some of our prior reported preliminary findings [12–14] from the studies underway at the CSIR on the development of versatile custom 17-4 PH MIM feedstocks. The

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current article reports on a comprehensive comparison of the static properties of the 17-4 PH materials prepared at the CSIR and those 17-4 PH MIM material properties reported by others in literature, the materials standard specifications for as-MIM 17-4 PH [15,16], 17-4 PH material properties specified in the material (powder and feedstock) suppliers' datasheets.

To make the comparison more meaningful, only the as-sintered 17-4 PH MIM properties are considered from at least fifty prior studies. The material properties of wrought 17-4 PH are included for completeness of the manuscript. This current report neglects the influences of the powder and feedstock characteristics, injection moulding and sintering parameters on the static mechanical properties (tensile, hardness, density and shrinkage) of the metal injection moulded 17-4 PH stainless steel materials.

2. Materials and Experimental Methods

Three starting 17-4 PH stainless steel powder materials, the -45 μ m Grade 630 (supplied by Praxair Surface Technologies), the -5 μ m and the -15 μ m (both supplied by Epson Atmix Corp) were used for this study. The morphologies of the starting metal powders are reported in Figure 1 while a summary of the powder particle size distributions and chemical composition have been reported elsewhere [12,14].



Figure 1. The SEM micrographs of the starting 17-4 PH stainless steel powder materials $-5\mu m$ (a), $-15\mu m$ (b) and $-45\mu m$ (c) at 1000x magnification [12,14].

2.1. Feedstock preparation and injection moulding

The MIM feedstocks were prepared by mixing 17-4 PH stainless powder materials with a proprietary wax-polymer binder system developed at the CSIR as explained elsewhere, see Ref.s [7,8,12,14]. The binder system consists of paraffin wax, low-density polyethene, polypropylene and stearic acid proprietary grades. The solids loading (volume ratio of the powder to the total volume of the powder and binder) used for the study ranged from 55 to 65 vol%. The feedstocks were granulated and then injection moulded into the standard 'dog-bone' tensile test specimen according to ISO 2740 specifications (see Figure 2(a)) at 140°C using a 40-tonne ARBURG Allrounder 270U 400-70 injection moulding machine. The optimization of the injection moulding process parameters (injection temperature, speed and pressure and mould temperature) in our previous work [7,8].

2.2. Debinding and sintering

Debinding of the green components was performed in two steps; i.e. solvent debinding in *n*-heptane (Merck) at 60 °C for 24 hours followed by thermal debinding at 550°C for 1 hour in a Carbolite tube furnace under a controlled atmosphere of argon gas flowing at 1.0 L/min. The debinding process parameters have been reported on before, see Ref.s [7,8]. The sintering of the debound components was also performed in two steps; a pre-sintering stage at 1000 °C for 1 hour in the tube furnace (flowing Ar. gas) followed by final sintering in a high-vacuum furnace. The final sintering step was performed at 1300 °C for 3 hours. A Xerion cold-wall high-vacuum furnace with molybdenum shield

packs and tungsten heating was used. The dimensions of the produced as-produced, solvent debound, and the as-sintered specimen are illustrated in Figure 2(b).

2.3. Post-sintering evaluation

The sintered samples were evaluated for density, microstructure, pore morphology, hardness and tensile properties. The density of the sintered samples was determined according to the immersion method (Archimedes' principle) as outlined in ASTM B311. The microstructure and pore distribution were determined using a Leica DMI500 M optical microscope. Apparent hardness measurements were carried using an automated Vickers microhardness tester (FM-700) as outlined in ASTM-E384. The tensile testing was conducted as per ISO 2740 standard on the INSTRON[™] Servo Hydraulic 1342 test instrument at a rate of 0.5 mm/min.



Figure 2. the (a) design drawing of the as-sintered 'dog-bone' test specimen as per ISO 2740 standard and (b) photographs of the produced as-moulded (A), solvent-debound (B) and sintered (C) tensile test specimen as reported previously in Ref. [12]. The dimensions shown are all in millimetres.

3. Results and Discussions

The processing of 17-4 PH via MIM is now fairly documented; however, a recent review published by German (during the preparation of this article) also submits that despite the widespread popularity of the 17-4 PH alloy, data on the sintered properties 'that can be expected' is still limited [3].



Figure 3. (a) the influence of feedstock solids loading and starting bimodal powder materials on the measured as-sintered density and (b) the typical optical micrograph of the as-sintered specimen

3.1. The microstructure and as-sintered densities of the sintered MIM 17-4 PH materials

The effects of the solids loading and using bimodal feedstocks on the sintered density is shown in Figure 3(a). The sintered density was shown in Figure 3(a) to increase with the increase in solids

loading of the feedstock. At low feedstock solids loading, the starting bimodal powder materials appear to have adversely negative effects on the sintered density while the effect is significantly positive at higher feedstock solids loading. These observations together ratify foundational PM tenets that the sintered density can be enhanced through the formulation of either highly loaded feedstocks or bimodal feedstocks, without necessitating longer sintering times or at higher sintering temperatures.

Figures 3(b) is included only to exhibit the typical microstructure observed from the 17-4 PH MIM specimens. Tentatively, the microstructural constituents of the shown dual-phase as-sintered are the delta-ferrite (light grey) and the martensitic (dark grey) phases – the dark regions are oxide inclusions and spherical pores. The microstructure is consistent with previously reported findings [2,3,17,18].

3.2. The mechanical properties of the as-sintered MIM 17-4 PH materials

The experimentally measured 17-4 PH as-sintered MIM mechanical properties resulting from the tensile tests (*MIM CSIR*) are displayed in Figure 4 along with the comparative (i) material properties specified in the powder and feedstock material suppliers' datasheets (*MIM Pwd* and *MIM FS*), (ii) assintered MIM mechanical properties reported in the literature (*MIM Exp*), (iii) material properties reported from various minimum standard specifications (*MIM Std*), and (iv) the mechanical properties 17-4 PH wrought material reported also in the literature properties (*Wrought*). From the author's previous work low feedstock solids loadings (e.g. 55 vol. %) and coarse starting 17-4 PH stainless steel powder materials (e.g. -45 μ m) tend to typically exhibit experimentally measured 17-4 PH mechanical properties [12] when compared to the material properties specified in the material suppliers' datasheets.



Figure 4. A comparison of the 17-4 PH material ultimate tensile strength (UTS), yield strength (YS), percentage elongation at failure (Elongation), and the measured apparent hardness (Hardness). MIM CSIR refers to the as-sintered MIM 17-4 PH mechanical properties; MIM Pwd and MIM FS - material properties specified in the powder and feedstock material suppliers' datasheets, respectively; MIM Exp - properties reported prior in the literature; MIM Std - properties reported from various minimum standard specifications; and Wrought - the literature reported mechanical properties 17-4 PH wrought material.

Available mechanical properties data for wrought 17-4 PH stainless steels materials is generally higher by up to 10 25 % compared to the 17-4 PH as-sintered MIM properties experimentally measured in this study. Reasons cited by Schade *et al.* [19] include the presence defects as a result of sintering (retained pores and grains, and residual carbon impurities), chemical composition deviations due to (Cr, Cu and Ni) elemental loss via evaporation during sintering and the inclusion of impurities (O, C, and N) [3], and but chiefly precipitation hardening during the fabrication of wrought 17-4 PH stainless steels materials typically increases the properties by as much as 15 to 20 %. While on the face of it is trivial, the fact that PM processes such as MIM seek to substitute wrought 17-4 PH stainless steels materials in various applications strongly necessitates this comparison.

The current article makes use of over thirty prior studies available in the literature reporting on the mechanical properties of the as-sintered MIM. The large scatter in the literature reported mechanical properties is understandably due to numerous reasons including a heterogeneity of the starting material sources, and differences in the processing conditions as lately pronounced by German [3]. Likewise, the large scatter in this study's experimentally measured mechanical properties can also be attributed to the numerous MIM feedstocks prepared and used. As clarified by Coleman *et al.* for MIM AISI 4605 materials [20], the huge variations depicted in Figure 4 from the tensile tests from the author's experimental work and properties reported in the literature is a testament that a wide range mechanical properties are feasible from the 17-4 PH alloy.

The experimentally measured as-sintered 17-4 PH MIM from this study are generally comparable to the minimum standard specifications for 17-4 PH MIM materials and the mechanical properties specified by various suppliers (powder and MIM feedstock) in data sheets, and the prior-reported MIM 17-4 PH material properties available in the literature.

4. Concluding Remarks

A comparative study of the CSIR metal injection moulded materials against the as-sintered MIM material properties according to the ASTM standard specification for MIM materials, prior-reported 17-4 PH material properties available in the literature, as well as 17-4 PH wrought and as-cast material properties were presented in this paper.

While the sintered density of the CSIR 17-4 PH material is generally lower than expected, the mechanical test results show that properties are comparable with industry ASTM standard specification. In addition, the processing of 17-4 PH stainless steel using the CSIR-developed MIM feedstocks and processing led to properties typically within those obtained when the parts were processed via prior-reported MIM studies. It is also evident from this study that low feedstock solids loadings (e.g. 55 vol. %) and coarse starting 17-4 PH stainless steel powder materials (e.g. -45 µm) tend to typically exhibit experimentally measured 17-4 PH mechanical properties.

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