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INJECTION MOULDING OF A BIMODAL SIZE DISTRIBUTION 316L STAINLESS STEEL POWDERS

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Abstrak. Bagi mengurangkan kos bahan mentah untuk proses pangacuan suntikan logam (MIM), bahan teracu yang diperbuat daripada serbuk logam keluli tahan karat dengan nisbah serbuk logam kasar dan halus yang berlainan telah diselidiki. Bahan suapan tersebut disediakan dengan menggunakan sistem bahan pengikat komposit yang terdiri sebahagian besarnya polietilina glikol (PEG) yang berlainan berat molekulnya dan emulsi polimetil metakrilat (PMMA) yang seterusnya disuntik ke dalam bentuk segiempat tepat dengan suhu muncung suntikan logam pada 140°C. Hasil ujikaji menunjukkan kekuatan bahan teracu meningkat secara linear dengan peningkatan nisbah serbuk halus di dalam campuran dan optimum pada 30 wt.% dan seterusnya berlaku penurunan dengan peningkatan nisbah serbuk halus. Peningkatan serbuk logam halus di dalam campuran juga dapat meningkatkan ketumpatan akhir dan kekerasan bahan tersinter. Kadar penyingkiran polietilina glikol semasa proses pengurasan air meningkat dengan penurunan nisbah serbuk logam halus yang mana menunjukkan bahawa serbuk logam kasar memberi kesan ketara ke atas proses penyingkiran bahan pengikat.

Kata Kunci: pengacuan suntikan logam, polietilina glikol, polimetil metakrilat, pengurasan air, pengsinteran

Abstract. In an effort to lower the cost of raw materials of metal injection moulding (MIM), a moulded part made of a different ratio of coarse and fine stainless steel powder mixtures, ranging from 10 to 50 wt.% of fine powder were investigated. The feedstocks were prepared using a composite binder system which consist of a major fraction of polyethylene glycol (PEG) with different molecular weight and a minor fraction of polymethyl methacrylate (PMMA) emulsion and subsequently moulded into a rectangular shape at the nozzle temperature of 140°C. The results show that the green strength of the moulded part increased linearly with the increased of the ratio of fine powder to an optimum of 30 wt.% and decreased slightly with the increased of coarse/fine ratio. An increase in the fine powder in the mixture, improved the final densification and hardness of the sintered parts. The rate of PEGs removed during water leaching substantially increased with the lower ratio of fine powder, which suggest that the coarse powder has significant influence on the removal of the binder.

Keywords: metal injection moulding, polyethylene glycol, polymethyl methacrylate, water leaching, sintering.

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1.0 INTRODUCTION

Metal injection moulding (MIM) is a rapidly developing technique that provide an alternative method for mass production of small, complex shapes and provide cost effectiveness. Currently the products receive a great deal of attention for their high performance properties. MIM process involves feedstock preparation from a suitable metal powder and binder, injection moulding of the part, removal of the binder by solvent or thermal method and finally sintering process to the final density [1].

An understanding of the powder characteristics is of fundamental importance in this process because it affects the properties of both the feedstock and the final product. Generally, in selecting the powder for MIM, it must be able to achieve high packing density that resists distortion in the as-moulded and debinded states, while being responsive to mixing, moulding, debinding and sintering. Fine powder is preferred as an ingredient for feedstock for MIM because such powder gives better mouldability and enhanced sintering. In addition, the smaller particles exhibit a desirable increased in compact strength during debinding that decreases the dimensional loss in processing [2].

Much of the published work on MIM in stainless steel powder was conducted using fine powders. [3–5]. However, such powder is more expensive and as alternative coarser and cheaper grades of powder are used in injection moulding process. The technical challenge is then to improve the rheological characteristics by using a relatively coarse and fine powder blends, while maintaining comparable sintered densities to those obtained with the use of fine powders. Meeting this challenge will not only result in improving the integrity of MIM part, as far as the presence of flaws introduced during moulding stage, but will also result in a more cost effective use of the available metal powders.

In this study, two types of stainless steel powder, one with a median of ~24 μm in diameter (coarse powder) and the other with a median of ~13 μm in diameter (fine powder) were employed. The respond of the feedstock made with different ratio of the coarse to fine powder to the green strength, debinding and sintering process at a given solid loading was attempted. This study is a continuation of a previous investigation [6–7] in which injection moulding of coarse 316L stainless steel powder with a composite binder system was evaluated. The emphasis was on achieving the highly sintered density with the low cost of processing.

2.0 MATERIALS AND METHODS

In this study, gas atomised powder with the median of ~24 mm in diameter supplied by Manganese Bronze Components designated as “coarse powder” and the fine powder with a median particle size of ~13 mm in diameter supplied by Osprey Ltd was used. The chemical composition of both powders is given in Table 1. The weight ratio of the coarse to fine powders were controlled at 90/10, 80/20, 30/70, 60/40, and

Table 1 Chemical composition of coarse and fine powders

Type	Elements, %							
	C	Si	Mn	P	S	Cr	Ni	Mo
24 μm	0.090	0.32	0.80	0.041	0.016	16.40	12.40	2.31
13 μm	0.017	0.69	1.32	0.030	0.002	17.19	13.82	2.77

50/50 and the solid loading was kept constant at 65 vol.%. A 24 hours-tumbling process was performed for each batch in order to get a homogenous mixture.

A two component binder system was adopted in this study which consist of a combination of various molecular weight of PEGs (600, 1000, and 1500) as the major component (85%) and finely dispersed PMMA emulsion as a minor component (15%). Initially, the metal powder was mixed with the PMMA emulsion to form a homogenous paste. The PEGs were melted and poured into the paste, which was then mixed thoroughly. The mixture was dried in the oven for 1 hour.

Injection moulding was performed using a pneumatically driven plunger type machine. The barrel was electrically heated using a heater jacket. The nozzle temperature was kept at 140°C and the injection pressure was 46 MPa. In order to improve the homogeneity and to reduce the amount of entrapped air, the feedstocks were extruded before moulding and then chopped into small pieces. The chopped pieces were re-fed and then injected through the nozzle into the mould cavity. Simple, rectangular bar-shaped test specimens with dimensions of $5 \times 5 \times 55$ mm were moulded. The resulting green compact was characterised in term of transverse rapture strength using a three point bending test.

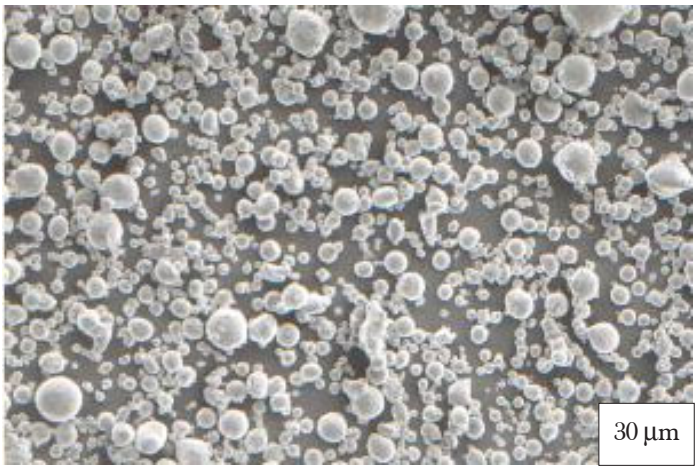
Debinding was performed in two stages; water leaching to remove the PEGs and thermal pyrolysis to remove the PMMA. Green specimens were immersed in distilled water held at a temperature of 60°C for 4 hours. The leached specimens were then dried in an oven at 50°C overnight to completely remove the remaining water. To determine the rate of PEGs removal as a function of time, the green specimens were cut into test specimens of about 15 mm long, and then leached at 60°C at various times, ranging from 15 to 300 mins and the weight loss was determined by weighing the dry samples.

For thermal pyrolysis, the leached specimens were put into the alumina tube in which the surrounding space was filled with alumina powder to avoid distortion of the specimens with the heating rate of 3°C/min to 400°C and held for 30 minutes. The specimens were then sintered in the same furnace as thermal pyrolysis in argon atmospheres. The heating rate employed was 5°C/min to 1360°C, holding for 2 hours. The densities of the sintered specimens were measured by water immersion method and the porosity was measured by image analyser. The hardness of the sintered specimen was determined by microvickers with the load of 100 gf.

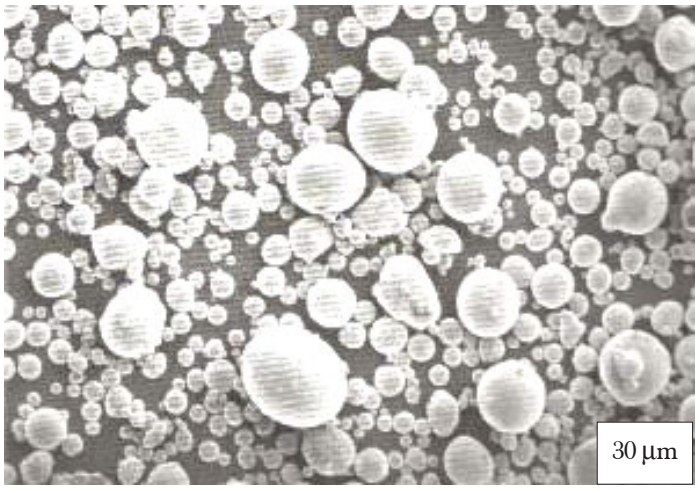
3.0 RESULTS AND DISCUSSION

3.1 Powder Characteristics and Feedstock Preparation

The shape of the particles and their surface textures of fine and coarse 316L stainless steel powders are shown in Figures 1a and b for the coarse and fine powders respectively. All the particles were approximately spherical and a large proportion had smooth surfaces. The spherical shape will result in high packing density which is required for injection moulding.



(*a*)



(*b*)

Figure 1 Scanning electron micrographs of the stainless steel powders, (*a*) fine powder and (*b*) coarse powder

For feedstock preparation, when the emulsion is mixed with the 316L stainless steel powders, the fine PMMA particles are easily dispersed between the powder particles. When the solution of the molten PEG are mixed in the mixture of powder and PMMA, they are obviously uniformly dispersed on a very fine scale. No segregation of the binder components was observed. This suggests that the mixing carried out was effective.

3.2 Moulding Behaviour and As-Moulded Strength

All the feedstocks were easily injected at 46 MPa with a temperature of either 135°C for a mixtures containing 10 and 20 wt.% fines or 140°C for those containing 30 to 50 wt. % fines. The increase in temperature follows from the fact that feedstocks made with finer powder have higher apparent viscosities [1].

The mixing of the fine and coarse powders should improve packing density efficiency [8]. Beginning with the coarse powder, the specific volume of the mixtures initially decreases as fine powders are added to fill the unoccupied voids between the large particles. Eventually, the quantity of fine powders is insurplus since all of the voids are filled. Thus, further additions will force the coarse powders apart which can no longer improve the packing density. However, in the present study all the mixtures prior to moulding had the same solids content and hence should have had essentially the same green density apart from minor differences which could occur from the presence of voids.

The as-moulded strengths for bar made with the different mixtures are shown in Figure 2. It shows that the maximum strength observed for the 70 wt.% coarse 30 wt.%

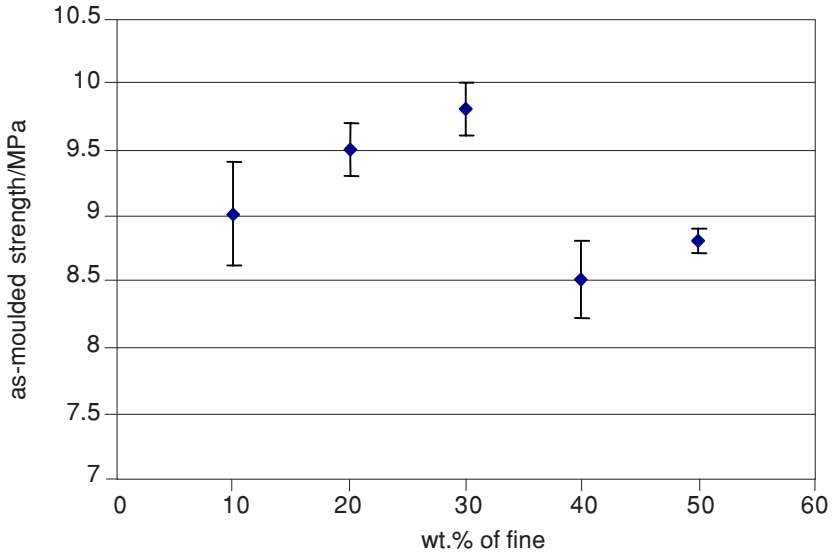
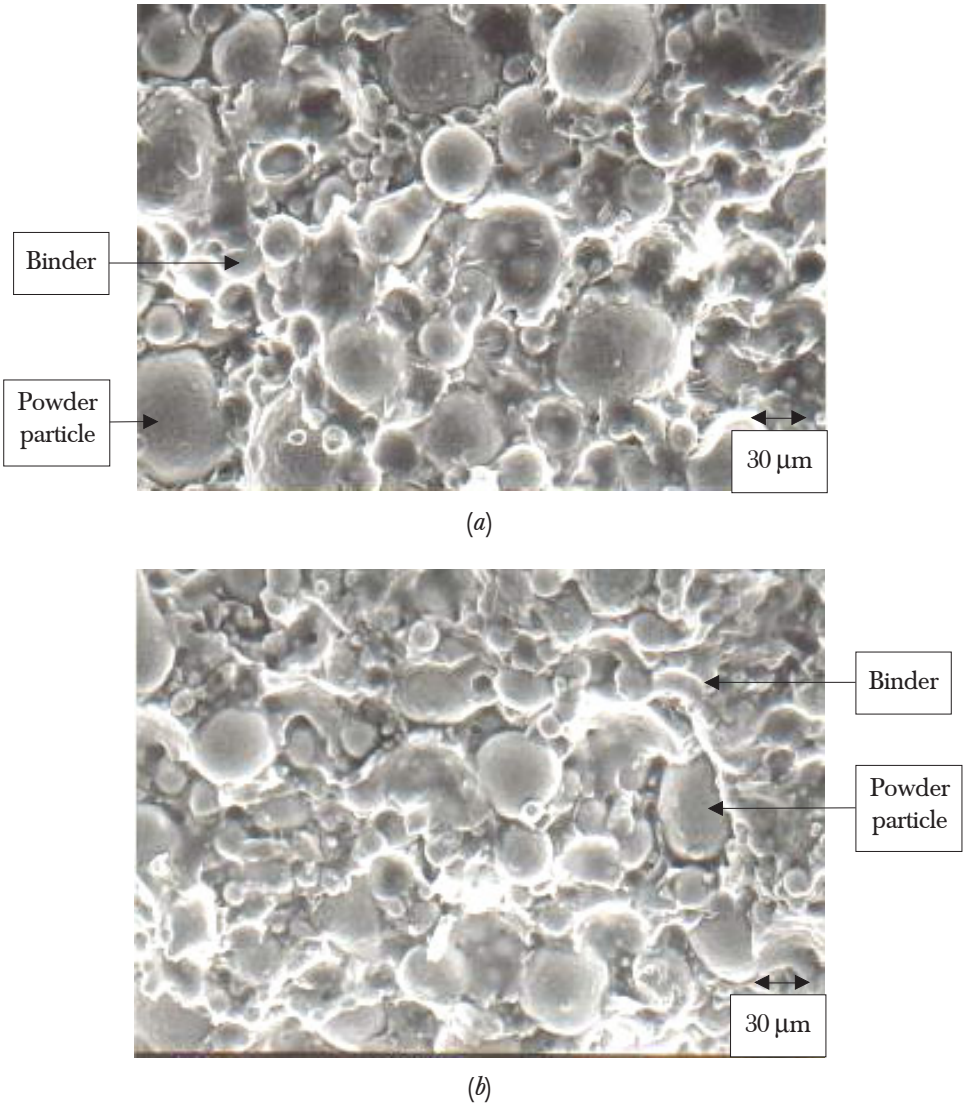


Figure 2 The as-moulded strengths of mouldings made with different amounts of coarse and fine powder

fine mixture, which suggests that particle packing density significantly affects the strength. Further increased in fine powder (up to 40 wt.%) clearly reduced packing density, resulting in lower as-moulded strength.

Figures 3a and b show the fractured surfaces of the moulded specimen made from 20 wt.% and 40 wt.% fines respectively. It can be seen that, the binder fills practically all the interstitial spaces between the powder particles. The densities of the as-moulded



Figures 3 Scanning electron micrographs showing the fractured surfaces of a moulding. The binders fills practically all the interstitial spaces between the powder particles, (*a*) 20 wt.% fine and (*b*) 40 wt.% fine

(green) bars are given in Figure 4. The average values of three bars are plotted along with the maximum and minimum values. These results did not show any significant correlation between the densities achieved with the wt.% of fines used. However, all the values are substantially higher than the theoretical density for void-free feedstock calculated from the batched weights of the components. The nominal solid content was 65.5 vol. %, however, the solid content calculated for a density of 5.67, which is the average value for all the bars, is approximately 67.2 vol.%. It is thought that the increase in solid content may due to the evaporation of the PEGs during hot mixing and hot extrusion prior to moulding.

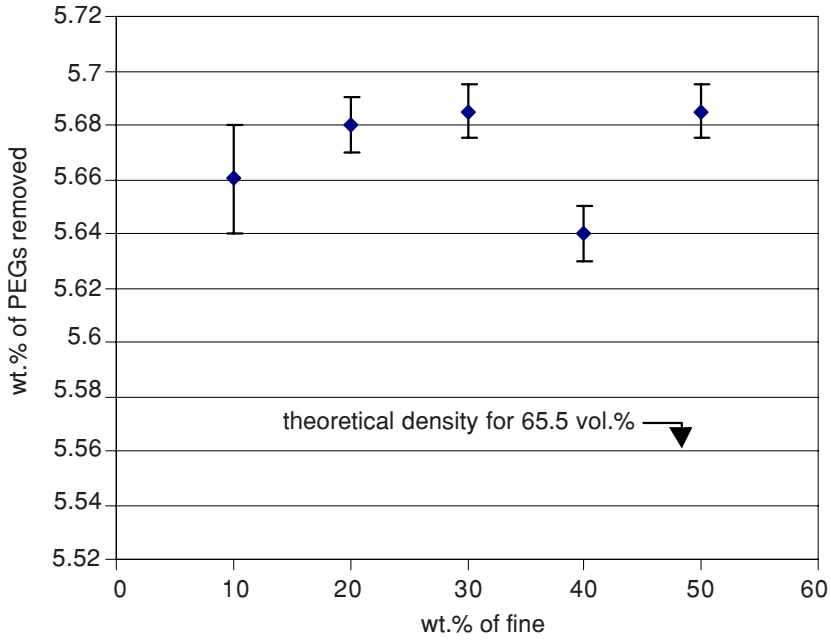


Figure 4 The densities of the as-moulded (green) bars for the different mixtures

It is proposed that during moulding the shearing of the feedstock at the mould surface causes the particles in this region to pack more densely. This allows binder to separate at the mould/feedstock interface, which facilitates the flow of the feedstock into the mould. On cooling, the more densely packed surface layer of the particles will shrink less than the bulk of the feedstock. Material is forced under pressure into the mould to compensate for the shrinkage and this will result in the overall as-moulded density to be higher than the feedstock value.

3.3 Leaching and As-leached Strength

Based on the results obtained in earlier part of the study [9, 10], water leaching which was carried out for 5 hours at 60°C and followed by drying for 1 hour in an oven at

50°C prior to thermal pyrolysis, the as-leached samples retained their shape and neither swelled nor surface blemishes were observed. Figure 5 shows the PEG weight loss percentage as a function of leaching time for the different mixtures of coarse and fine powders. These results indicate that removal of the PEGs from the mouldings become progressively slower with decreasing overall mean particle size.

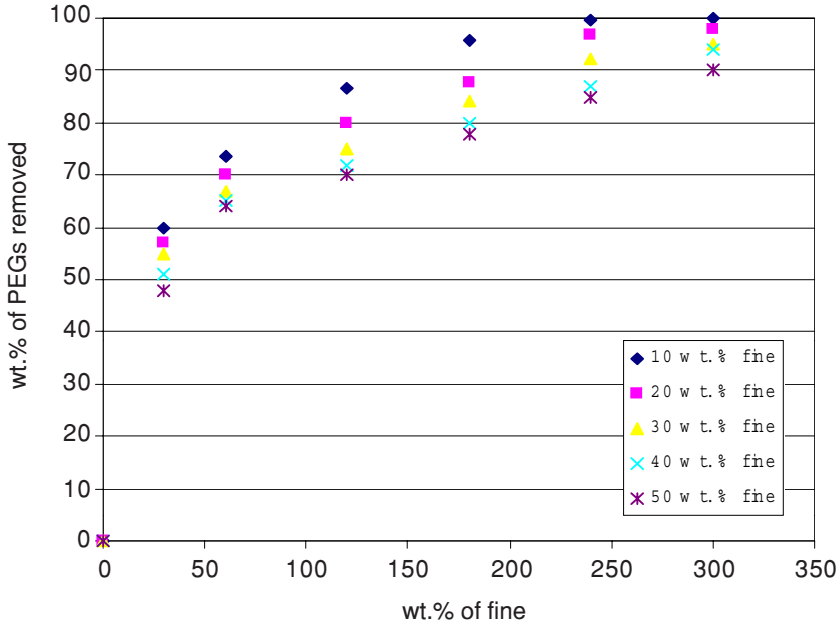


Figure 5 The percentage weight loss of PEGs as a function of leaching time for different mixtures of coarse and fine powders

Figure 6 shows the as-leached (brown) strength for different mixtures. Increasing the fine particles in the blends increased the strength of the as-leached parts. The average as-leached strength of the moulded parts increased from 0.57 to 0.75 MPa as the wt.% of fine particle increased from 10 wt.% to 50 wt.%. These are more than a factor of ten lower than the as-moulded values, but the bars were still strong enough to be handled. The strengths show a steep increase in value when the content of fines is increased from 20 to 30 wt.%. This possibly reflects the suggested denser surface particle packing. However, the strength continues to increase as the content of fine increases. The as-leached strength arises from the PMMA bonding the particles together. These results suggest that this bonding depends on the surface area of the particles in the moulding, which increases with increase in the content of fines.

3.4 Sintering Behaviour

Figure 7 shows the densities of the injection moulded compacts as a function of the wt.% of fine powder in the mixtures sintered at 1360°C for 4 hours in argon. Not

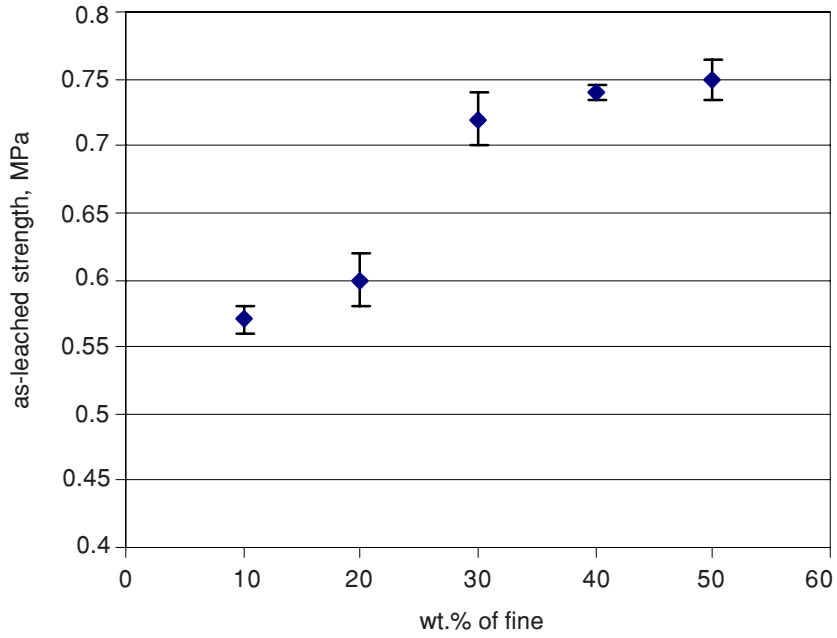


Figure 6 As-leached strength with different ratio of fine powder

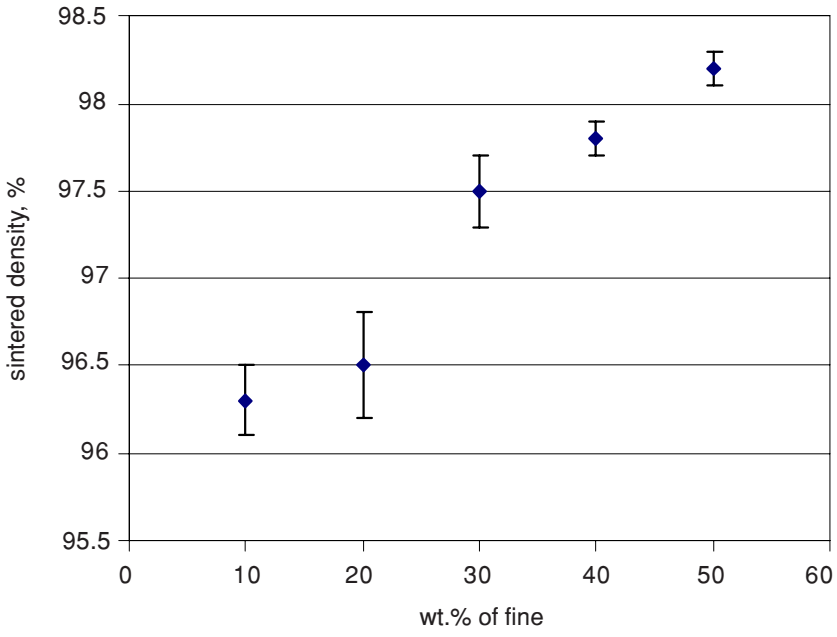


Figure 7 Relative sintered densities of bars sintered at 1360°C for 2 hours in 5%H_2+95%N_2 as a function of the content of fine powder

surprisingly, increasing the wt.% of fine particles resulted in higher sintered densities. When the wt.% of fine increased from 10 to 50 wt.%, the average sintered density increased from 96.3% to 98.3% of the theoretical maximum value (7.9 g/cm^3). The progressive addition of fine powder clearly improves densification probably because of the increase in surface area and hence an increase in driving force for sintering. German [8] reported that mouldings made with bimodal powder mixture, the highest packing occurred for a mixture consisting of 70 wt.% coarse and 30 wt.% fine powder. Unfortunately, he found that the sintering densification was poor for such a mixture and the optimal densification was observed between 80 to 100% of fine powder. The linear shrinkage was observed to range between 11 and 13% and increasing the content of fine powder increased the shrinkage of the sintered specimens primarily because of the increased densification.

The improved sintering activity affects the mechanical property behaviour. Increasing the fine fraction of mixtures, increased the hardness of the sintered samples because of the reduced porosity as shown in Figure 8. The optical micrographs show the microstructure of the sintered specimens at 20 wt.% and 40 wt.% of fine powder. The hardness of the mouldings sintered in a 5%H_2/95%N_2 atmosphere increased from 150 Hv to 180 Hv when the fine powder in the mixtures was increased from 10 wt.% to 50 wt.% as shown in Figure 9.

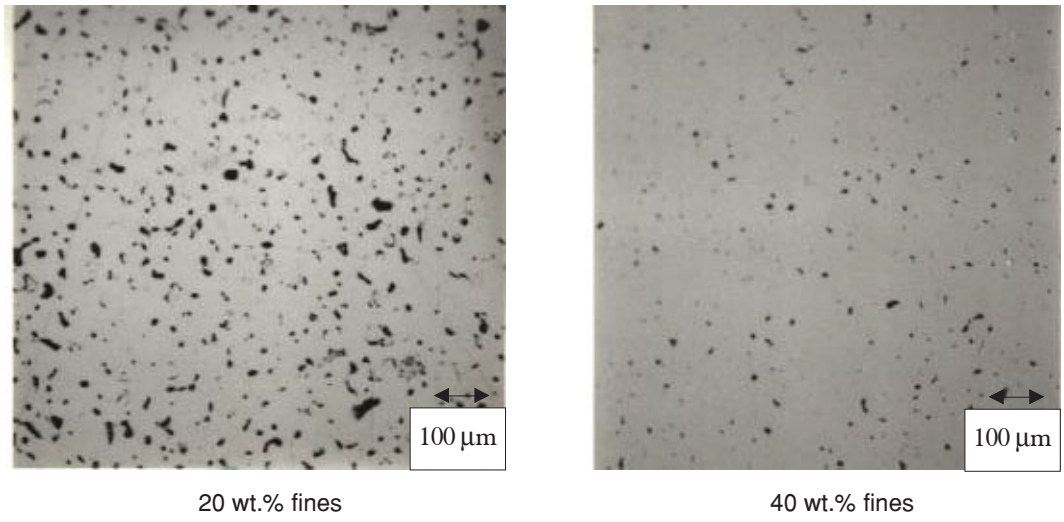


Figure 8 Optical micrographs of the sintered specimens showing the microstructure of the specimens at different wt.% of fine powders

4.0 CONCLUSIONS

The influence of particle size, on the green and sintered densities in 316L stainless steel-PEG/PMMA blends was investigated. An increase of the wt.% of fine powders in

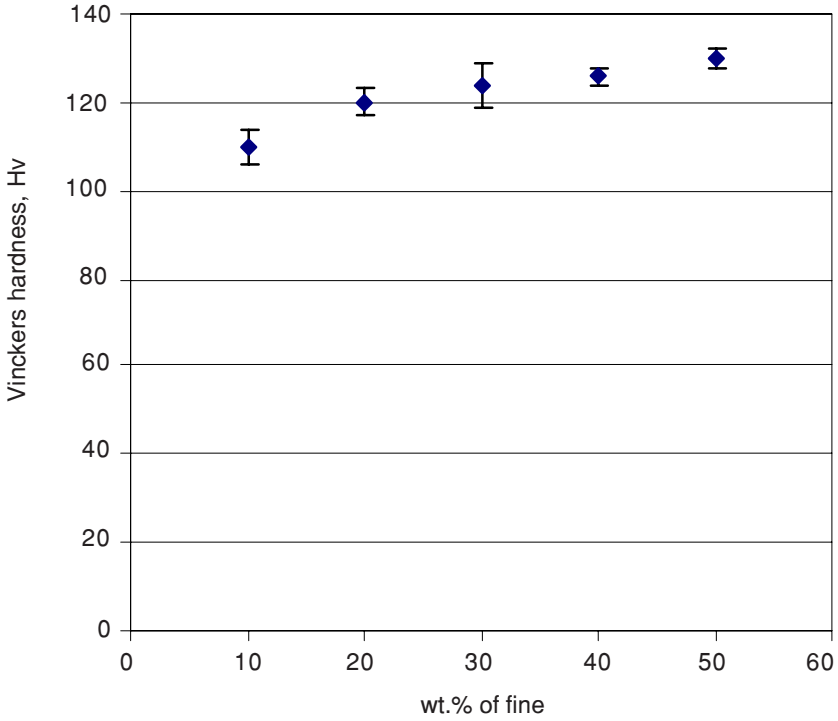


Figure 9 Vickers microhardness for sintered 316L stainless steel compacts based on various fractions of fine and coarse powders

the mixture up to 30 wt.% increases the as-moulded strength and the green density as well. Further increase in wt.% of fine provides no improvement in the as-moulded strength and green densities and this may result from poorer particle packing efficiency. An increase in the fine powder in the mixture, improved the final densification and hardness of the sintered parts. It can be concluded that the lower cost combination of powders such as 50 wt.% fine and 50 wt.% coarse may be satisfactory for parts, which do not require full density. Application requiring a controlled porosity condition could also use such powder mixtures.

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