AMM012

The 19th Conference of mechanical Engineering Network of Thailand
19-21 October 2005, Phuket, Thailand

Preparation of 316L-Al_2O_3 Composite

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Abstract

The 'as-received' austenitic stainless steel 316L powder (Coldstream, Belgium), premixed with 0.8% Acrawax + 0.2% lithium strearate, was sieved into two fractions, which were in the ranges of 75-106 μm and 32-45 μm, respectively. Sieved fractions were mixed with the volume ratio of 78.54% of the 75-106 μm fraction and 17.45% of the 32-45 μm fraction. The mixture of sieved fractions was designated as ''as-sieved' 316L'. The 'as-sieved' 316L was mixed with 2% and 4% by volume of Al_2O_3 powders, with particle size less than 20 μm. The powders, 'as-received' 316L, 'as-sieved' 316L, 'as-sieved' 316L + 2% Al_2O_3 and 'as-sieved' 316L + 4% Al_2O_3 were compacted into tensile test bars with green density of 6.50 ± 0.05 g/cm^3. The green compacts were then sintered at 1300 °C for 45 minutes in pure hydrogen. It was found that addition of Al_2O_3 resulted in slight decrease of sintered density, ultimate tensile strength, yield strength and elongation, but slight increase of hardness. Decrease of strength was attributed to sintering prohibition by Al_2O_3 particles. This might be improved by enhancing wettability of 316L on Al_2O_3 particles. The 316L-Al_2O_3 composite, with reduced weight and increased hardness, might be applied as a tooling material.

Keywords: Powder Metallurgy, 316L-Al_2O_3 composite

Introduction

Powder metallurgical method has some advantages for fabricating of particulate reinforced metal matrix composites because it offers material and energy saving as well as dimensional accuracy. Fabricating method may include simple steps, such as powder mixing, pressing, debinding and sintering. Powder metallurgy of stainless steels has been attracting numerous interests from both research and development sectors as well as industrial ones [1, 2]. This is attributed to stainless steel benefits, which include high strength and good corrosion resistance. However, there is an effort to produce a material, with combined properties of high strength, good corrosion resistance and sufficient hardness, for tooling applications. Stainless steel matrix composites are therefore ideal candidate materials.

Some works have been carried out to produced particulate reinforced stainless steel matrix composites [3]. Stainless steels and tools steels were reinforced with particulate reinforcements such as Al_2O_3, TiC, Cr_2C_3 and TiN [4]. The hot isostatically hipped materials showed that the incorporation of a relatively low volume fraction of ceramic particulate reinforcements significantly increased the wear resistance of the steel matrices, without deteriorating the corrosion properties. However, the material exhibited reductions in the tensile strength, ductility and toughness. Reinforcement type and amount and sintering atmosphere showed influence on properties of 316L matrix composites [5]. Yttria alumina garnet (YAG) reinforced 316L matrix composites, prepared by either solid-state-sintering or supersolidus sintering, improved hardness compared to that of sintered 316L material [6].

Experimental Procedure

The '***as-received***' austenitic stainless steel 316L powder (Coldstream, Belgium), premixed with 0.8% Acrawax + 0.2% lithium strearate, was sieved into two fractions, which were in the ranges of 75-106 μm and 32-45 μm, respectively. Sieved fractions were mixed with the volume ratio of 78.54% of the 75-106 μm fraction and 17.45% of the 32-45 μm fraction. The mixture of sieved fractions was designated as '***as-sieved***' 316L. The 'as-sieved' 316L was mixed with 2% and 4% by volume of Al_2O_3 powders, with particle size less than 20 μm. It was expected that powder particle packing of the 'as-sieved' 316L + Al_2O_3 would be a tri-modal packing as shown in Fig. 1. Characterisation of the powders, 'as-received' 316L, 'as-sieved' 316L, 'as-sieved' 316L + 2% Al_2O_3 and 'as-sieved' 316L + 4% Al_2O_3, was carried out to obtain flow rate, apparent density and morphology. The powders were compacted, using a uniaxial press, into tensile test bars (TTBs) with green density of 6.50 ± 0.05 g/cm^3. The green TTBs were then sintered at 1300 °C for 45 minutes in pure hydrogen. Dimensional changes of the TTBs, along different dimensions shown in Fig. 2, during compaction and sintering were measured. Physical and mechanical properties of the sintered TTBs were measured and compared among the different sintered materials. Microstructures were also observed using optical microscopy and scanning electron microscopy (SEM).

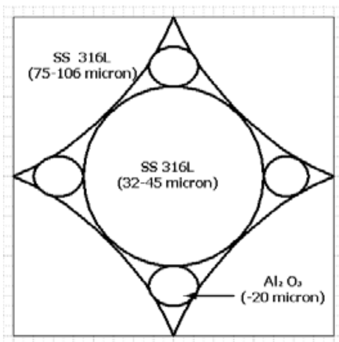
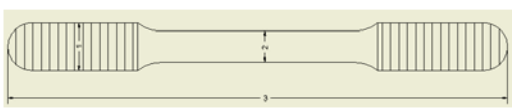


Figure 1. Tri-modal particle packing of the 'as-sieved' 316L + Al_2O_3 mixture.



1 = grip
2 = gauge
3 = length

Figure 2. Dimensions on a TTB measured for dimensional change.

Results and Discussion

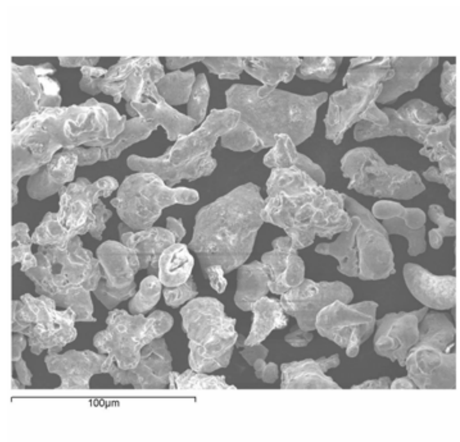
(1) Powder characterisation

The 316L stainless steel powder flow rate was altered when it was modified (Table 1). The ''as-sieved'' 316L powder, obtained by remixing of powders with particle size ranges of 75-106 μm and 32-45 μm, flowed faster than the ''as-received'' 316L powder. This indicates that powder particle rearrangement has some effect on powder flow. The optimised powder particle rearrangement that maximizes flow rate is out of scope of this investigation. However, it is recommended here that this issue is worth for further investigation because powder flow information is needed for P/M industry.

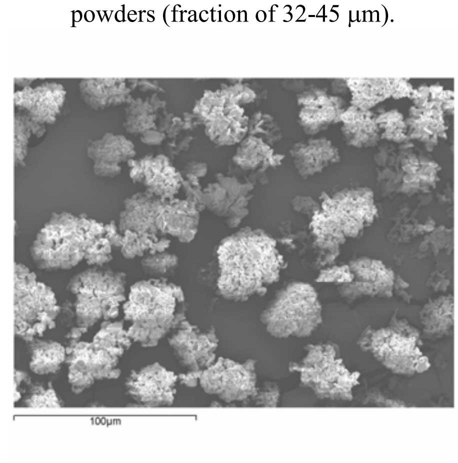
Addition of fine Al_2O_3 to the ''as-sieved'' 316L decreased the flow rate. This is attributed to interparticle-friction increase due to the presence of Al_2O_3 particles with irregular surfaces, (Fig. 3 (b) and (c)), which are able to cause particle interlocking with irregular shape 316L powder particles (Fig. 3(a)). Apparent density indicates particle packing character of the powders. The ''as-sieved'' 316L powder improved apparent density indicating better particle packing. Addition of Al_2O_3 to the ''as-sieved'' 316L decreased apparent density, which was attributed to two factors, such as poor particle packing and the presence of low-density Al_2O_3 particles.

Table 1 Flow rate and apparent density of different powder types.

Powder	Flow rate (Sec/50g)	Apparent density (g/cm^3)
'as-received' 316L	36.69	2.75
'as-sieved' 316L	33.72	2.77
'as-sieved' 316L + 2% Al_2O_3	34.00	2.68
'as-sieved' 316L + 4% Al_2O_3	36.67	2.60



(a) Morphology of sieved 316L stainless steel powders (fraction of 32-45 μm).



(b) Morphology of Al_2O_3 powders (particle sizes less than 20 μm).

Figure 3. Morphology of powders

(2) Dimensional change during compaction

During uniaxial compaction, the powder particles are squeezed to form a compact in a rigid die. The load exerting to the powder particles may be divided into two types, namely axial and radial loads. Both loads cause residual stresses, which are stored in the compacts. Radial expansion of the compact after being ejected from the die is called "spring back". Spring back is the phenomenon related to residual stress releasing. Spring back of the green TTBs was calculated from changes of their dimensions with respect to those of the die.

Plots of spring back against powder types for the green TTBs are shown in Fig. 4. Spring back along the TTB gauge showed the highest value, followed by those along grip and length, respectively. Magnitude of the stress is probably dependent on dimension of the green TTBs. In case of perfect powder filling, higher stress is generated along shorter dimension. It is thus expected that the residual stress along the TTB gauge compacts is higher than that along other dimensions.

Spring back seemed to increase with increasing Al_2O_3 content. Spring back is probably related to material deformation. When the softer 316L powder particles are squeezed against one another and against harder Al_2O_3 powder particles, some of 316L powder particles deform elastically. The elastically deformed particles recovered to their original shape and size after the exerting pressure is removed during ejection of the green TTBs from the die.

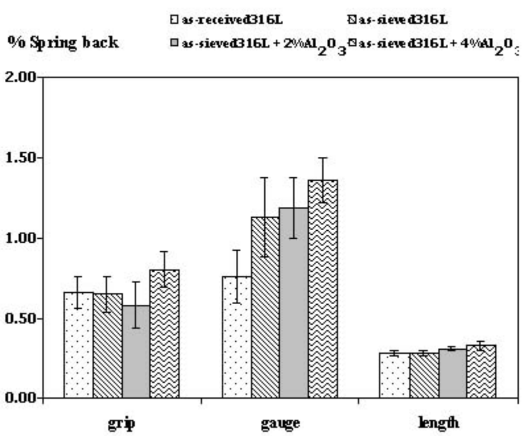


Figure 4. Spring back of the green TTBs made from different powder types.

Addition of coarse Cu powder particles was effective to reduce spring back along the gauge of a TTB made from 409L stainless steel powders [7]. Coarse Cu powder particles substitute the 409L stainless steel powder particles at some sites. It was supposed that the radial load, exerting to the powder particles, was less than yield strength of the 409L particles. During compacting step, the substitutional coarse Cu particles were squeezed by 409L particles, which were in turn pressed by a radial load. Plastic deformation of the substitutional coarse Cu particles was expected to occur. The plastic deformation would reduce elastic deformation at points of contact between the 409L particles. Small amount of elastic deformation would therefore cause small level of spring back.

(3) Sintered density and dimensional change during sintering process

The TTBs made from different powder types showed different sintered density. The 'as-sieved' 316L TTBs showed sintered density higher than that of the 'as-received' 316L ones. Addition of Al_2O_3 to the 'as-sieved' 316L resulted in decreased sintered density. Sintered density was related to various factors such as green density, points of contact between particles, materials transport phenomena during sintering process. dimensional change during P/M processing steps, compaction and sintering.

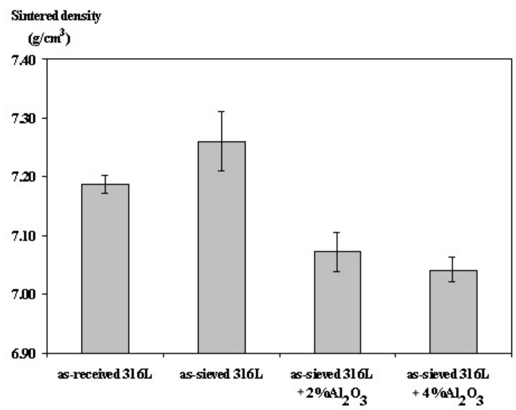


Figure 5. Sintered density of the TTBs made from different powder types.

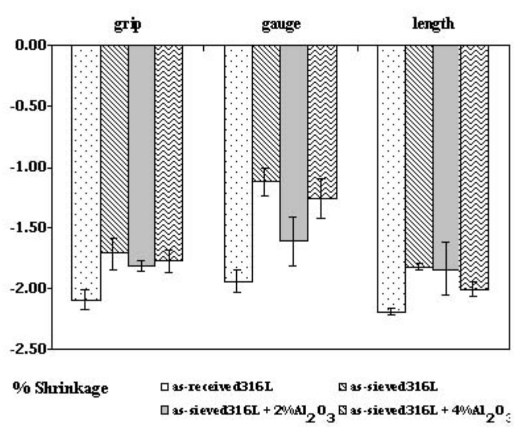
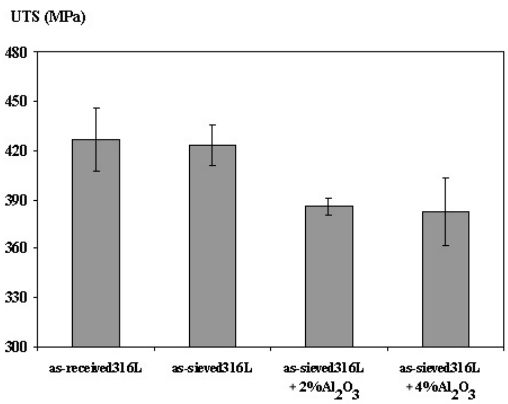


Figure 6. Shrinkage of the sintered TTBs made from different powder types.

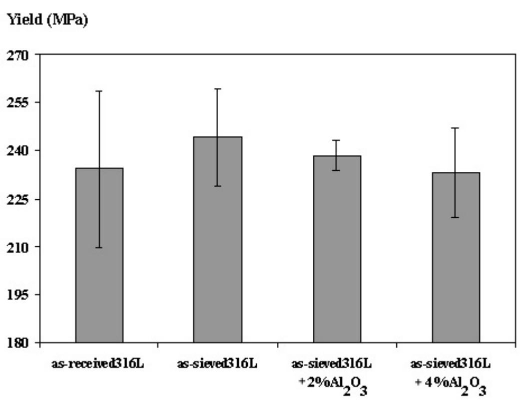
(4) Mechanical property

Most values of mechanical properties (UTS, elongation and hardness) of the sintered 'as-sieved' 316L TTBs were close to those of the sintered 'as-received' 316L TTBs. The only property that these two sintered materials showed different values was yield strength. The difference in yield strength is not understood yet.

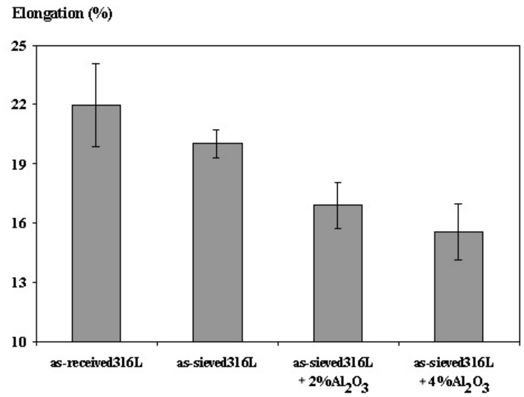
Addition of Al_2O_3 particles to the 'as-sieved' 316L resulted in decreased tensile properties (UTS, yield strength and elongation) of the sintered TTBs (Fig. 7(a)-(c)). This may be attributed to sintering prohibition by foreign Al_2O_3 particles. Adhesion or wettability between the 316L and Al_2O_3 particles may also be one of the prime factors affecting bonding between matrix and reinforcements. Microstructure observation of the sintered TTBs would clarify the tensile property decrease. Introduction of hard Al_2O_3 particles into 316L matrix resulted in increasing hardness, which is perhaps the only benefit obtained from the Al_2O_3-316L composites prepared in this study.



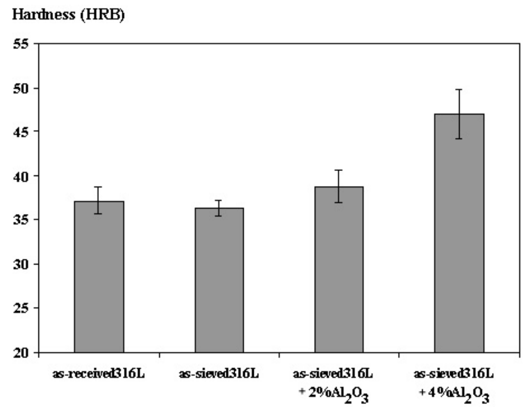
(a) UTS



(b) Yield



(c) Elongation



(d) Hardness

Figure 7. Mechanical Properties of the sintered TTBs made from different powder types.

5. Microstructural observation

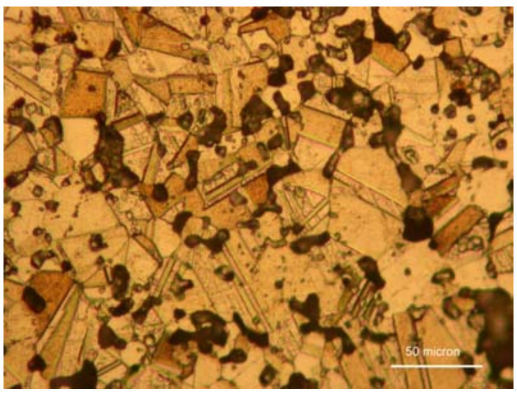
The sintered 'as-sieved' 316L TTBs showed microstructure consisting of smaller pore volume fraction and size (Fig 8 (b)) than those observed in the sintered 'as-received' 316L TTBs (Fig 8 (a)). The improved microstructure is attributed particle packing quality. Better particle packing, indicated by higher apparent density, leads to generation of more points of contact, which encourage densification phenomena by solid-state sintering.

Microstructures of the sintered 'as-sieved' 316L + Al_2O_3 TTBs (Fig. 8 (c)-(e)) showed pockets (in the case of non Al_2O_3 containing TTBs, the pockets are equal to pores) filled with Al_2O_3 particle aggregates along grain boundaries. Evidences of interparticle bonding formation between Al_2O_3 particles themselves and between 316L-Al_2O_3 particles, were hardly observed (Fig. 8(e)). No bonding between Al_2O_3 particles is due to a low temperature sintering process. No bonding between Al_2O_3-316L particles is attributed to poor wettability of metals on Al_2O_3 particles, since their electrons are tightly bound and their surfaces represent large discontinuities in charge [4].

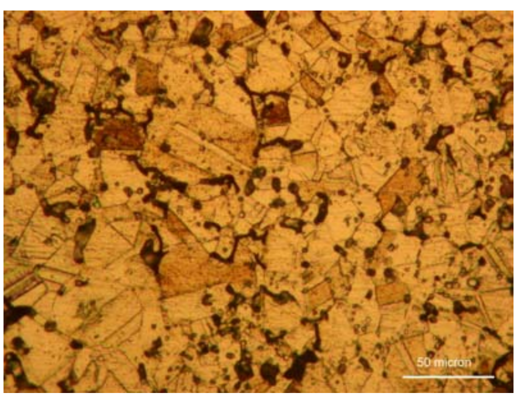
Decrease of tensile properties (UTS, yield strength and elongation) of the 'as-sieved 316L +

Al_2O_3 is clearly due to the following reasons. The first reason is related to poor Al_2O_3 particle distribution. Al_2O_3 particle aggregates could be easily observed in the pockets between 316L matrix (Fig. 8(e)). Poor reinforcement particle distribution in the matrix results in that the composite material loses some useful properties. The second is related to poor wettability between matrix and reinforcement. In the case of the Al_2O_3-316L composites prepared in this study, poor wettability obviously causes detrimental effects on tensile property.

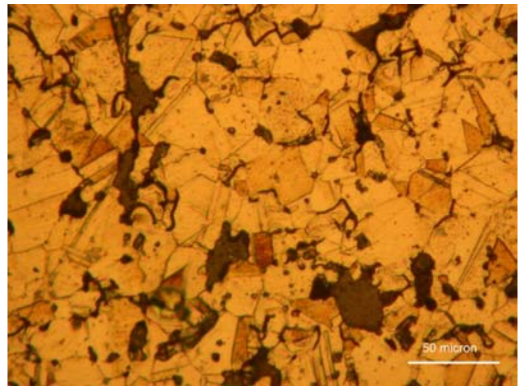
Due to poor properties obtained from the solid-state sintering of Al_2O_3-316L composites, it is recommended here that further improvement is needed to be carried out in the future. The potential techniques to improve the Al_2O_3-316L composite properties include supersolidus sintering, mechnical milling of the powders and liquid phase sintering.



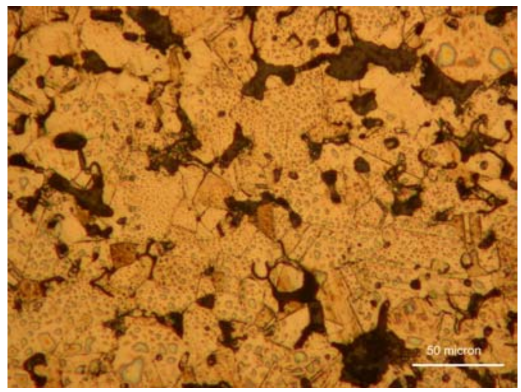
(a) Optical micrograph of the sintered ‘‘as-received’’ 316L TTB



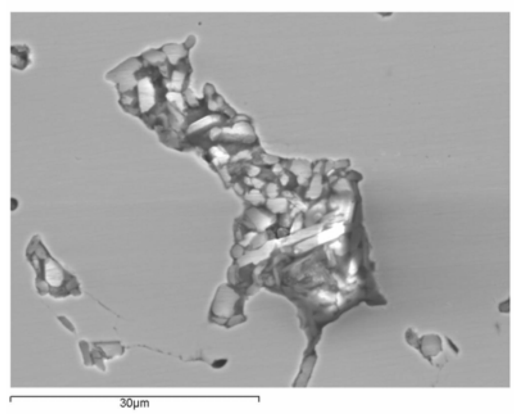
(b) Optical micrograph of the sintered ‘‘as-sieved’’ 316L TTB



(c) Optical micrograph of the sintered ‘‘as-sieved’’ 316L + 2% Al_2O_3 TTB



(d) Optical micrograph of the sintered ‘as-sieved’ 316L + 4% Al_2O_3 TTB



(e) SEM micrograph showing Al_2O_3-filled pockets in the Microstructures of the sintered ‘as-sieved’ 316L + Al_2O_3 TTBs

Fig. 8 Microstructures of the sintered TTBs made from different powder types.

Conclusions

Addition of Al_2O_3 resulted in decrease of sintered density, UTS, yield strength and elongation, but increase of hardness. Decrease of strength was attributed to sintering prohibition by Al_2O_3 particle aggregates. This might be improved by enhancing wettability of 316L on Al_2O_3 particles. Al_2O_3 particle distribution improvement may also be needed for preparation of the 316L-Al_2O_3 composite. Although the 316L-Al_2O_3 composite exhibits inferior tensile property compared to P/M 316L alloy, its reduced weight and increased hardness might be useful for an application as a tooling material.

Acknowledgment

The authors would like to express their sincere gratitude to Metal and Materials technology Center (MTEC) for financial support for this investigation.

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