Microstructure and Mechanical Properties of an Al-Si Alloy Consolidated by Spark Plasma Sintering

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ABSTRACT

The Spark Plasma Sintering (SPS) is a promising sintering technology to produce dense bulk pre-compacts from micro- or nano-structured aluminium alloys at lower temperatures and shorter sintering times. The densification behaviour and sintering response of an atomized Al-Si alloy sintered by SPS was investigated. A high density of bulk material with porosity less than 1% can be prepared by SPS with a temperature of 450°C, holding for 2-10min and a pressure of 170 MPa. In addition, the homogeneity of the microstructure across the diameter of the sintered samples are investigated and correlated with in-situ measurements of the temperature distribution within the samples. Residual porosity is mainly localised at the margin close to the wall of the die, corresponding to the lowest measured temperatures. The influence of the process parameters on the structure and tensile properties of subsequently hot extruded material was studied and compared to material based on hot pressed pre-compacts. The mechanical tensile properties of the SPSed compacts are significantly lower compared to that of the extruded material due to some residual porosity and the weakness of the joint between the initial atomized alloy powder particles. The extruded P/M material offers superior mechanical strength to the comparable compositions of known die cast piston alloys.

INTRODUCTION

Rapidly solidified P/M Al-Si alloys with higher content of silicon and transition metals are expected to be applied for automotive engine components such as pistons or connecting rods thanks to a low coefficient of thermal expansion, high wear resistance and excellent mechanical properties. Rapid solidification is an effective way to get a supersaturated solid solution, thermally stable dispersoids, metastable phases or amorphous phase, but limiting the thickness of the rapidly solidified structure, which is therefore available only within fine powders, thin ribbons or flakes. The challenge of producing a bulk product from such prealloyed powders is the need to perform a complete consolidation at the lowest possible temperature in a short time with preservation of the unique nano-/microstructure to the bulk material.

In this context, the recently developed field assisted sintering technology offers unique advantages in material fabrication compared to the conventional P/M techniques. It belongs to a class of sintering techniques that employ electric current to assist sintering [1], and Spark Plasma Sintering (SPS) is one of the most recent versions of these techniques [2-4]. Regardless the controversy to the presence/absence of sparking [5], important technological benefits such as short processing time, fewer processing steps, elimination of the need for sintering aids, and near net shape capability are unchallenged. In addition, the use of high heating rates and short dwell times can minimise grain growth, which often leads to improved material properties.

Several experimental studies [6-16] on the effect of pulsed current sintering on microstructure and mechanical behaviour of pure and alloyed aluminium powders demonstrated the capability of SPS in maintaining a very fine microstructures as prerequisite

for a excellent mechanical performance of the bulk material. The breakdown of the oxide layers on the aluminium powder particles is one of the challenges for successful sintering of aluminium. The available experimental results suggest, the SPS process can promote the elimination of the closed oxide layers and therefore promote the sintering by localised heating at the contact areas in combination with a sufficient pressure on the powder. In addition, the necessary degassing step for P/M aluminium should be also feasible in a short time by employing the sintering cycle to remove the adsorbed gas on the particles effectively. But, the measured tensile mechanical properties of aluminium compacts consolidated by SPS scatter between lower and comparable with that of an corresponding wrought or extruded compact [7, 11, 14, 15]. Therefore, it seems, that no comprehensive understanding regarding the influence of the SPS sintering process parameters and material characteristics exists today.

In this paper, a hypereutectic Al-Si alloy has been processed by SPS and subsequent hot extrusion to investigate the effect of relevant processing parameters. The effects of these parameters were investigated by means of microstructural analysis and tensile tests.

EXPERIMENTAL

The starting material was gas atomized powder sieved to <50μm of an Al-19Si-3,0(Fe,Ni)-2,5(Cu,Mg) alloy (supplied by ECKA Granulate GmbH, Germany) with a melting range of (530…680)°C and a density of 2,72 g/cm^3.

The pre-compacts were fabricated by hot pressing with a direct resistance heating for very fast heating using FCT-HP D 250 of FCT Systeme GmbH, Germany (Fig.1). The samples were heated by a pulsed electric current which flows through the punch-die-sample-assembly using a high current and low voltage. The current was forced through the powder charge by lining the inner sides of the steel die with a layer of an electrical insulator.

Figure 1. Aluminium billets (Ø 75mm, h 85mm, 99,3% theoretical density) consolidated by SPS using the FCT-HP D 250 of FCT Systeme GmbH, Germany.

All runs were executed in dynamic vacuum using one heating step from room temperature to the desired sintering temperature. After the desired holding time had elapsed, the power was shutt off and the assembly was allowed to cool down to room temperature. The pressure ranging between 170…230MPa was applied in two variants: 1. constant pressure for the whole sintering process, and 2. a low initial pressure of about 68 MPa was applied during the heating to 450°C and raised to the maximum value after several minutes (Fig. 2). The second processing variant should lead to a more pronounced degassing of the powder which is necessary for a successful compaction.
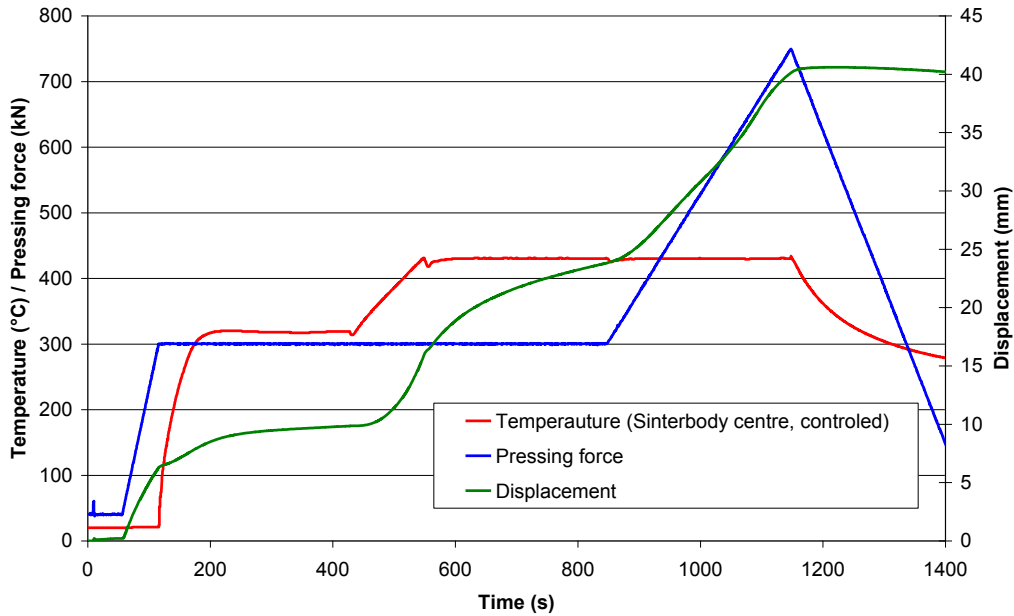
Figure 2. Time-temperature and time-pressing force dependance during the used Spark Plasma Sintering process. Additionally, the displacment of the puch electrodes is shown.

In some special experiments, additional thermocouples were placed inside the sinterbody to measure the temperature distribution during the SPS process [17].

For comparison, some pre-compacts were fabricated by hot pressing (HP) with an indirect resistance heating using HPW 315/400, manufactured by FCT Systeme GmbH, Germany. The samples were slowly heated in a cylindrical graphite die and a dynamic vacuum and subsequently consolidated at 450°C for 45min with a pressure of about 40 MPa under inert gas.

Subsequently, the sintered pre-compacts were deformed by direct hot extrusion at 400°C to rods of 25mm in diameter with a extrusion ratio of 10:1.

The bulk density of the composites was measured by using a method based on Archimedes' law and compared with the theoretical density. Microstructures of the sintered and deformed materials were studied by optical microscopy. Hydrogen and oxygen contents of the as-delivered powder and the consolidated specimens were measured by hot extraction with the LECO O/N-analyser TCH 600.

Tensile samples with a rectangular gauge area of $31mm^2$ and a gauge length of 35mm were machined from the SPSed pre-compacts and the extruded rods. Tensile properties were measured at room temperature using the ZWICK 1476-universal testing machine with a strain rate of $0,008s^{-1}$. The Young`s modulus was determined by using the resonance frequency method. The Brinell hardness tests (HB2,5/62,5) were done at room temperature with a EMCO tester M4U-250.

RESULTS AND DISCUSSION

The relative densitiy of the SPSed pre-compacts is about 99,3% of the theoretical density independent from the sintering schedule. Residual porosity is clearly visible within the compact close to die (Fig. 3a). The optical micrographs reveal a very fine microstructure of the pre-compacts, the silicon therein having an average particle diameter of not more than about 4μm. In addition, the microstructure shows partly undeformed nearly original spherical powder particles, but in well contact among each other.

a - close to the lateral shell

b - about 10mm from the lateral shell
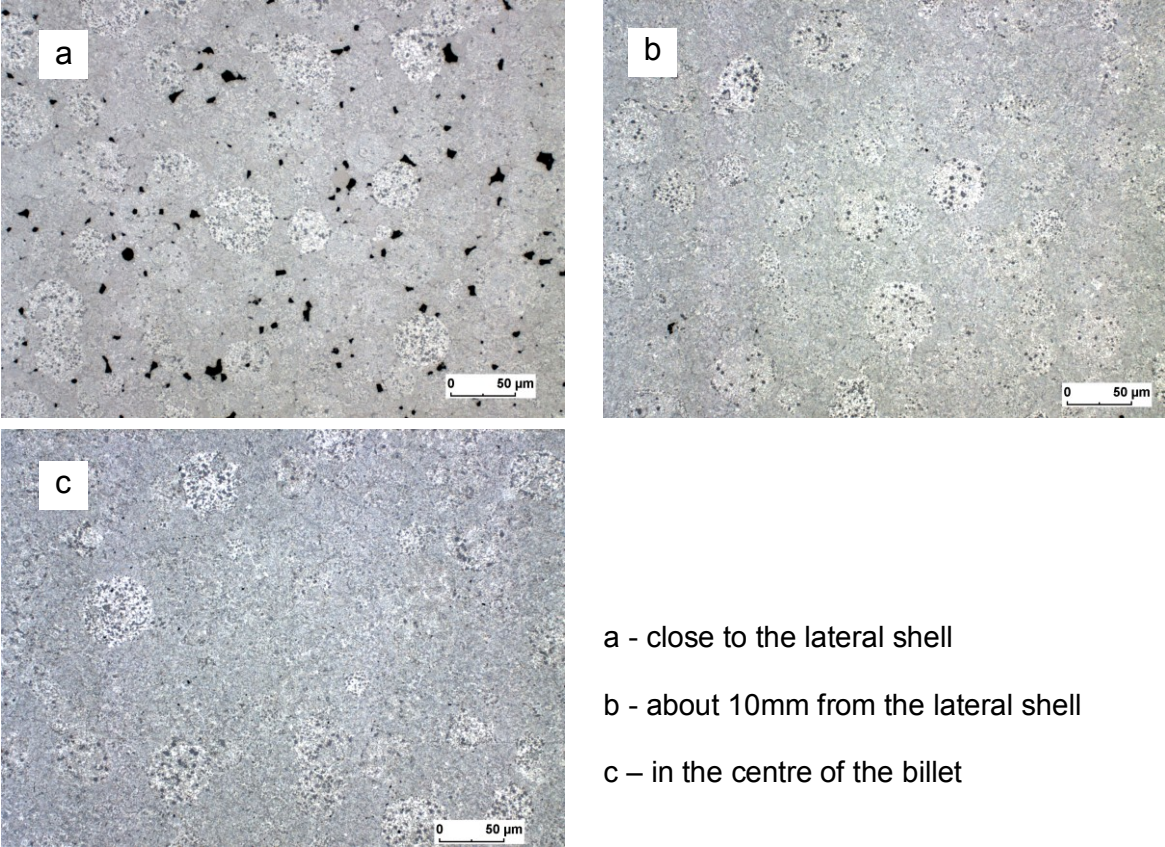
c – in the centre of the billet

Figure 3. Optical micrographs of cross-sections at different radial positions in the middle of a SPS processed billet.
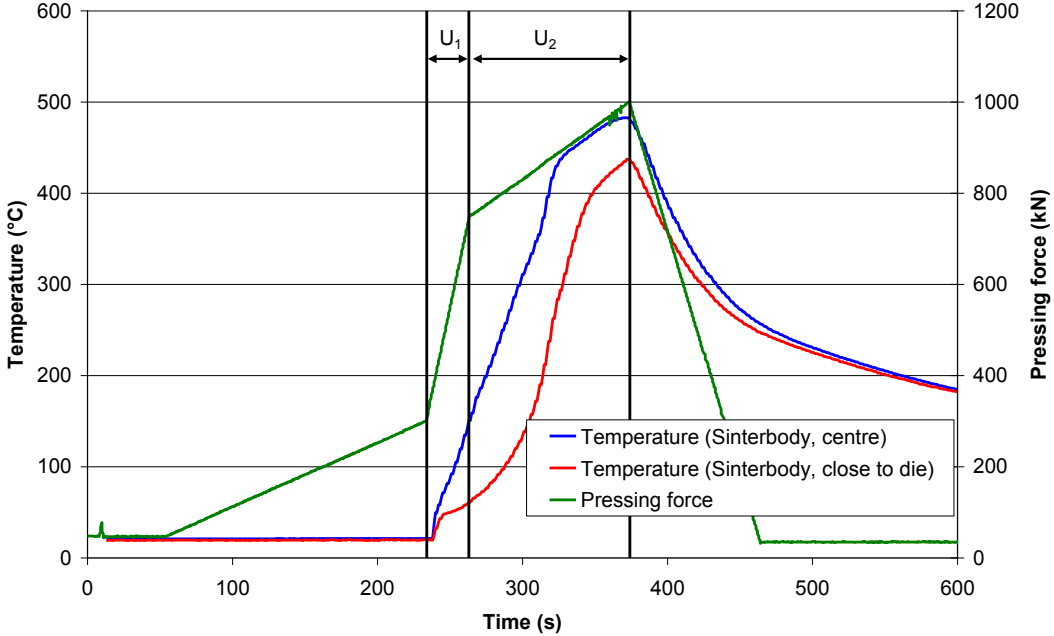

Figure 4. Time-temperature dependence inside the sinterbody during the Spark Plasma Sintering process. The measuring was done with external thermocouples, coming from the upper punch about 40 mm deep inside the sinterbody. One was placed in the centre of the sinterbody, one on the edge, about 10 mm away from the die wall.
The thermal analysis by using of additional thermocouples showed that the temperature at the centre of the sinterbody is higher than at the margin close to the wall of the die.

Especially a ring segment between 0 and 10 mm away from the die wall show significant lower temperatures compared to the inner part of the cylindrical disk. During the heating, the difference is decreased but in the thermal maximum, there is a temperature gradient with a $\Delta T = 44$ K between the measuring points in the centre and 30 mm away (Fig. 4). The origin for this cooling effect arises from the used pressing tool setup. The pressing die is not connected to the electrical circuit, and therefore its temperature is relatively low.

Figure 5 shows the microstructure at the central part of the extruded rods, exhibiting a retained fine and homogeneous microstructure, which is necessary to achieve a good mechanical performance of the consolidated material. In addition, some typical streamlines can be seen in the longitudinal section. The relative density of the extruded material is 100% theoretical density.
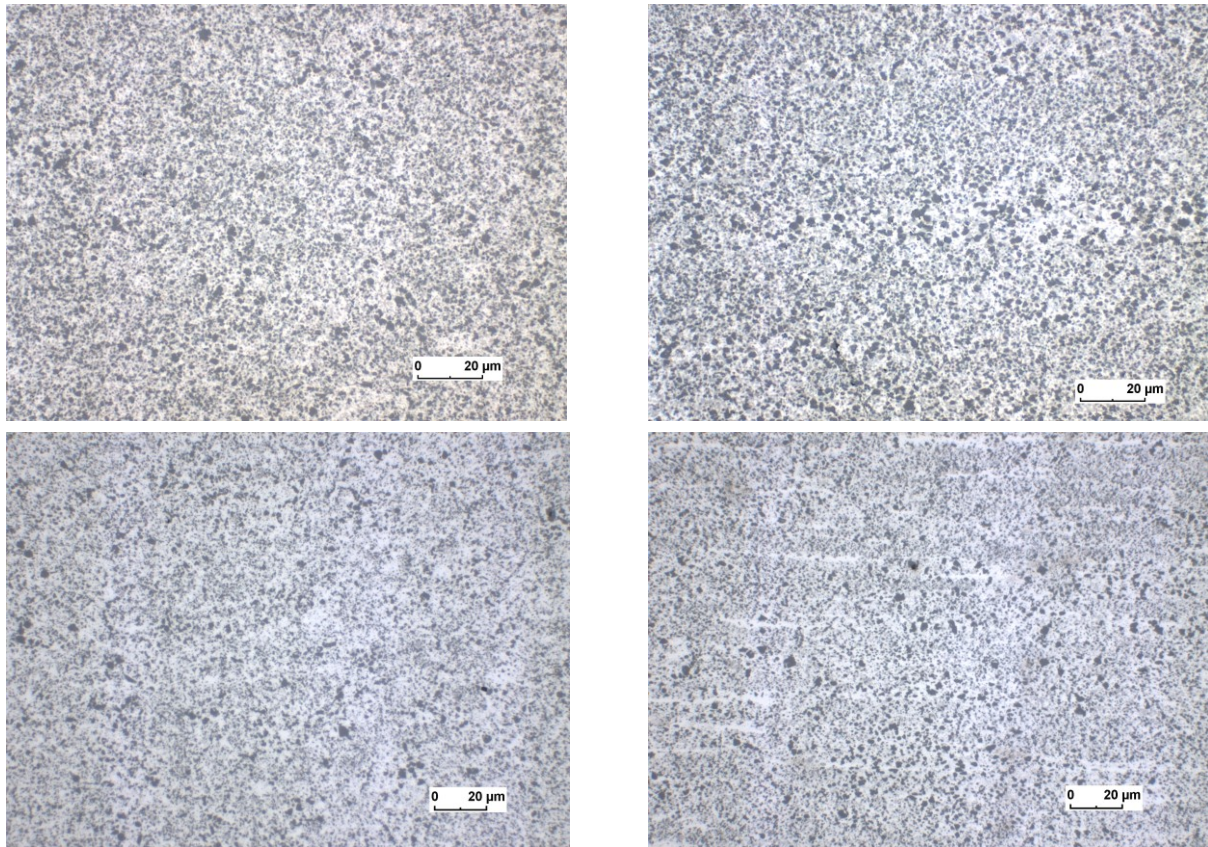

Figure 5. Optical micrographs of cross (left) and longitudinal (right) sections of a SPS/hot extruded rod (top) and a HP/hot extruded rod (below).

Table 1 presents a comparison of the mechanical properties of the materials fabricated by the SPS process and the combined consolidation, which is the combination of SPS with the direct extrusion. Although the hardness value of the SPSed compacts is higher than that of the extruded materials, the tensile properties are significantly lower due to some residual porosity and especially the weakness of the joint between the initial atomized alloy powder particles. This confirms the results given e.g. in [14]. The extruded P/M material offers superior mechanical strength to the comparable compositions of well known die cast piston alloys (e.g. MAHLE 138: Al-18Si-1,5Cu-1,3Mg-1,3Ni-0,5Fe). These good mechanical properties seems to be independently from the consolidation method of the pre-compacts. Therfore, the fast pressing techniques, e.g. Spark Plasma Sintering (SPS), offer advantages regarding shorter processing times and higher efficiency, compared to traditional hot pressing.

	Relative density in %	Brinell hardness HB 2.5/62.5	Young's Modulus in GPa	UTS in MPa	Elongation A in %
SPS 2	99.3	180	n.d.	105…215	0
SPS 1 + extrusion	100	157	92	435	1.5
SPS 2 + extrusion	100	171	92	455	1.3
HP + extrusion	100	160	92	430	1.6
MAHLE 138 [18]	100	90…125	83	230…300	0.5…1.5

Table 1. Comparison of the mechanical properties of the consolidated materials produced by different methods

It is well known, that adsorbed water on the aluminium powder surfaces not only causes an alumina barrier during sintering, but also inhibits the densification by generating hydrogen gas resulting in lower mechanical properties of the consoidated material. Therfore, the aluminium powder must be degassed before consolidation. Table 2 shows the measured hydrogen and oxygen contents of the different materials investigated.

	Hydrogen content in ppm	Oxygen content in ppm
Atomized powder	36	76
SPS 2	17	64
SPS 1 + extrusion	20	51
SPS 2 + extrusion	15	62
HP + extrusion	5	43

Table 2. Hydrogen and oxygen contents of the powder and the consolidated materials produced by different methods.

During all of the preformed consolidation methods a sufficient degassing was achieved resulting in reduced hydrogen contents of the pre-compacts and extruded material. Obviously, a better degassing was obtained by hot pressing because of the longer processing time. Further variations of the processing parameters are necessary for more understanding the degassing step during SPS.

CONCLUSIONS

Hypereutectic AlSi bulk pre-compacts with porosity less than 1% was fabricated by SPS at a temperature of 450°C, holding for few minutes and a pressure of about 170 MPa. Residual porosity is mainly localised at the margin close to the wall of the die, corresponding to the lowest measured temperatures. The mechanical tensile properties of the SPSed compacts

are significantly lower compared to that of the subsequently extruded material due to the weakness of the joint between the initial atomized alloy powder particles. The selected procedure of an in situ degassing step during the SPS results in reduced hydrogen contents. During a conventional hot pressing this degassing was more efficient because of the longer processing time. But, the resulting mechanical properties of the extruded material are not significantly influenced by these different levels of degassing. Therfore, the fast pressing technique Spark Plasma Sintering (SPS) offers advantages regarding shorter processing times and higher efficiency, compared to traditional hot pressing.

The extruded P/M material offers superior mechanical strength compared to compositions of well known die cast piston alloys due to the retained finer microstructures.

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