The Effects of Sintering Schedule on Alloy Powder ASC 100.29 Compacts Formed at Elevated Temperature

Z Hasan*, M A Ismail and M M Rahman
Department of Mechanical Engineering, Universiti Tenaga Nasional, Putrajaya Campus, Jalan IKRAM-UNITEN, 43000 Kajang, Selangor, Malaysia

*zaimah@uniten.edu.my

Abstract. This paper presents the outcomes of an experimental investigations on the effect of sintering schedule i.e. holding time and temperature to the final properties of alloy powder ASC 100.29 formed through uniaxial die compaction process at elevated temperature. A lab-scale powder compaction rig with uniaxial compression and installation of heater was designed and fabricated in order to produce green compact from powder mass at elevated temperature. Alloy powder ASC 100.29 (99.7 wt%) and lubricant, zinc stearate (0.3 wt%) were mixed mechanically for 30 min at a rotation of 30 rpm. The powder mass was compacted at 150°C via simultaneous upward and downward uniaxial compression load of 425 MPa. Defect-free green compacts were generated from the compaction and were subsequently sintered at 1000°C using a custom argon gas fired furnace at three different sintering rates, i.e., 5, 10 and 15°C/min for three holding times, i.e., 30, 60 and 90 min, respectively. The final products were characterized through their physical, electrical and mechanical properties and their microstructures evaluated. The results revealed that the sample sintered for a longer period of time, i.e. 90 minutes at the slowest sintering rate, i.e. 5°C/min obtained the better final characteristics.

1. Introduction
The rapid developments in powder metallurgy have led material processing route into a new phase especially in past few decades since these techniques offer alternative way to mitigate the deficiencies of the traditional methods of material processing such as forging, casting, stamping, forming and machining. Powder metallurgy process involved three main components, i.e., powder mixing, powder compaction into desired shape and sintering of the products generated from compaction [1]. Nowadays, the demand of the alloy in industries has increased due to its ability to improve the properties of the material in many aspects, i.e., physical, mechanical, chemical, electrical and thermal properties, thus capable of resisting damage at critical environment especially under high mechanical stress at higher temperature and in corrosive atmosphere, as well as becoming more versatile [2-4].

There are several methods available to produce alloy components but these methods are not economical as these methods require series of linked lengthy processes, hence the long production time, inefficient use of energy and more extra scrap losses produced. This is the especially evident when most of the alloys are transformed in billet form in the first place before undergoing various processing technologies to convert it into required alloy components [5]. Alternatively, utilization of pre-alloyed powder through warm powder compaction process has been widely chosen to produce alloy components where the process begins with feedstock preparation involving formulated pre-alloyed powder and mixing before compacting the powder mass at elevated temperature into desired shape and to the final process which is sintering of green compacts in controlled atmosphere [6]. Alloy components equipped with high strength and near-net-shape can be achieved by applying this technique since it is relatively new although has been initiated in 1998 [6].
Several research activities are found on the warm compaction process which practically used iron ASC 100.29 powder as main powder constituent to form alloy components with other different elemental powders in order to fulfill the demands [7-9]. The results revealed that high quality green compact alloy components can be achieved through compaction at elevated temperature and pressing simultaneously from both upward and downward direction compared to cold compaction method. Green compacts generated with higher density and green strength are expected to have higher flexural, tensile and fatigue strength yet better dimensional tolerances [10]. The frictional force is unavoidable during compaction due to friction between the die wall and the powders as well as among the powder particles itself. Zinc stearate is used as lubricant to reduce the frictional force and to extend tool life. The optimum value of lubricant is important to generate high quality alloy components since it was able to influence the density of the product [11-12].

A lot of research activities are reported on green compact generation at elevated temperature but no thorough study on characterization of sintering schedule especially on near-net-shape alloy components production has been investigated. Sintering is a process of heat treatment on the green compact products conducted in controlled environment in order to complete a full cycle of powder compaction process. Sintering process tends to reduce the surface contact among particles powder after material transport has been initiated through change of pore geometry, particles growth and shrinkage of pore volume [13]. The attraction of atomic and molecular bonding in powder compacts during heat treatment allows the development of a solid coherent body and subsequently, increases the strength of particles bonding as well as the rate of the desired degree of bonding among the particles [14].

Sintering is controlled by three main parameters, i.e., sintering temperature, sintering rate (heating and cooling) and holding time. These three parameters can be manipulated in order to improve the microstructure and reduce the number of pores inside the samples. There are three phases in the sintering process which started with heating phase, then holding phase (high temperature phase) and lastly, cooling phase which is assisted by transport mechanisms, i.e., plastic or viscous flow, diffusion (surface, volume, and grain-boundary) and atoms evaporated on surfaces, thus affecting the recrystallization and densification [14]. Densification process can be interpreted through the relationship of the sintered density to the mass changes and the volume (dimensional) since they are directly related to each other [15].

Proper sintering schedule is important to the powder metallurgy process as sintering generally can improve the properties of green compact product in many aspects. Correct sintering schedules need to be identified in order to produce near net shape of final alloy powder ASC 100.29 product since the current practice only focuses on foundry process and mechanical alloying. Thus, the objective of this paper is to investigate experimentally the effect of sintering schedule to the alloy powder ASC 100.29 compacts formed at elevated temperature.

2. Materials and Methods

The experimental process was conducted by applying four consecutive steps, i.e., a) feedstock preparation, (b) compaction and shaping, (c) sintering, and (d) sample characterization. In this experiment, 99.7 wt% of alloy powder ASC 100.29 manufactured by Höganäs AB was utilized and the balance was zinc stearate powder as lubricant. Alloy powder ASC 100.29 and zinc stearate were mixed mechanically for 30 min at a rotation of 30 rpm. The mixed powder mass was filled into cylinder shaped die cavity and compacted by using T-15 compaction rig at 150°C through axial loadings from both upward and downward direction at 425 MPa simultaneously to generate defect-free green compact with sufficient strength. Sintering process was executed by using a custom argon gas fired furnace (Model: HT3-1400-SIC, S/N: LT007) and all defect-free green compacts were sintered at a constant sintering temperature of 1000 °C at different sintering rates (5, 10 and 15°C/min) and different holding times (30, 60 and 90 min), respectively.

Both the green samples and sintered products were characterized for their physical and electrical properties. Afterwards, sintered products were subsequently characterized for their mechanical properties and their microstructures were analysed. The density of each sample was calculated by using the mass and volume (cylinder) of the sample. The density of the product was divided by the pore free density in order to find the relative density of the product. The electrical resistivity was calculated by measuring the resistance (ohm) using a digital multimeter (Brand: Fluke, Model: Fluke 115, S/N: 28341190ws) and the bending strength was measured by using three point bending test machine (Brand:
Instron, Model: Instron 3365, S/N: SAA61569) based on the standard (ASTM E290-09). The microstructures of the sample were evaluated by analysing the image of the fractured surface via scanning electron microscopy (Brand: JEOL, Model: JSM- 6010PLUS/LA).

3. Results and Discussion

The results of relative densities for final products are recorded in figure 1 after sintering via 5°C/min, 10°C/min and 15°C/min of sintering rate for 30 minutes, 60 minutes and 90 minutes of holding time, respectively. Relative densities of whole samples escalated after all green compacts were sintered using these parameters. The highest relative density was acquired by the samples sintered at 15°C/min of sintering rate for 90 minutes holding time. The samples sintered at prolonged holding time are exposed with longer high temperature phase which is the most important phase in sintering process. The maximum sintering temperature was continuously applied here based on the holding time and thus, the powder particles have fully reacted to initiate inter-particle bonding where the grains and necks started to grow and this has resulted to a decrement in size of pores inside the samples [16]. As for the sintering rate, 10°C/min and 15°C/min seemed to have the capability to provide higher relative density when the holding time is prolonged. Slower sintering rate might have affected the duration of sintering process where the time will be increased especially during heating and cooling phase.

After sintering, the powder compacts might experience either shrinkage or swelling due to various circumstances for example green compact density, the size of particle, heating rate, processing temperature, material compatibility and other factors [17-18]. In figure 2, the result indicated that the final products have gone through shrinkage after sintering schedule was applied on green compacts. Sample sintered at 15°C/min of sintering rate for 60 minutes of holding time is recorded as having highest percentage of volumetric shrinkage. Sample sintered using 15°C/min of sintering rate seemed to have both highest and lowest percentage of volumetric shrinkage at different holding times which are 60 minutes and 30 minutes, respectively. Higher percentage of volumetric shrinkage is not recommended although it is able to give high relative density near-net-shape products. The effects of sintering parameters to the volumetric shrinkage are assisted by the number of pores left from the zinc stearate that disappeared upon full burning during sintering.

The electrical resistivity of the sample has been influenced by sintering parameters resulting to the final products to have obtained lower electrical resistivity compared to green compacts. The electrical resistivity of the final products is shown in figure 3 and the result indicated that all samples have almost similar value except for 3 samples which have higher resistivity value. Highest electrical resistivity is obtained by the sample sintered at 5°C/min of sintering rate for 90 minutes of holding time. Theoretically, lower electrical resistivity caused electrical conductivity to be higher due to the current flowing inside the sample without having difficulty [19].

Bending strength is defined as the ability of the material to resist deformation under load and acted as a key factor for mechanical stability. Bending strength of the sample sintered under different sintering parameters is shown in figure 4. Highest bending strength is obtained by sample sintered at 10°C/min
of sintering rate for 90 minutes of holding time and the lowest is acquired by sample sintered at 10°C/min for 30 minutes. Based on the results, longer holding time is capable to produce higher bending strength of final products. Bending strength of final products might be affected by the number of pores left by zinc stearate since it was able to initiate crack propagation among interconnected pores when load is applied.

The microscopic images of final products at 1500 of scale magnification are shown in Figure 5. The result indicated that 5°C/min of sintering rate acquired a better quality of microstructure compared to 10°C/min and 15°C/min of samples since the grain boundary area have been reduced due to grain growth and simultaneously reduced surface area. Thus, the number of pores have been reduced and only consisted of small sized pores but at certain area, the small pores are connected to each other to develop a link of interconnected pores. The grain growth in alloy powder mass under slow sintering rate may avoid development of micro-cracks due to the thermal shock and subsequently, reduced the load bearing capability of the products [20].

4. Conclusion
The effects of sintering schedule in producing cylindrical shaped products from alloy powder ASC 100.29 compacts formed at 150°C was investigated. The results revealed that higher relative density, bending strength and electrical resistivity with moderate percentage of volumetric shrinkage can be achieved by using 90 minutes of holding time. As for sintering rate, 5°C/min can be considered to be suitable for ASC 100.29 alloy in order to generate microstructure with good inter particle bonding with reduced number of pores and higher electrical resistivity in final products.
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