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Research paper

An Experimental Investigation on the Effect of Sintering Temperature and Holding Time to the Characteristics of Fecual Powder Compacts Formed at Elevated Temperature

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Abstract

This paper presents the experimental investigation on the effect of sintering schedule to the final properties of FeCuAl powder compacts formed at elevated temperature through a lab-scale uniaxial die compaction rig. Iron (Fe) powder ASC100.29 was used as a main powder constituent and mixed with elemental powders which are copper (Cu) and aluminum (Al). The weight percentage of powder mass was divided into four, i.e., iron (91.7 wt%), copper (7.5 wt%), aluminum (0.5 wt%), and zinc stearate (0.3 wt%) as lubricant. All the powders were mixed through mechanical blending at a rotation speed of 30 rpm for 30 min. The mixed powder mass was compacted at 150˚C by 425 MPa of axial loading from upward and downward simultaneously. Subsequently, the defect-free green compacts were sintered under controlled argon gas atmosphere at three different sintering temperatures, i.e., 800˚C, 900˚C and 1000˚C for 120 min, 150 min and 180 min, respectively at constant sintering rate of 10˚C/min. Afterwards, the sintered samples were characterized for their physical properties, electrical properties, mechanical properties and their microstructures were evaluated. The results revealed that higher flexure stress was acquired by sample sintered at 1000˚C for 120 min and their microstructures were found to be better, i.e., the particles were bonded perfectly.

Keywords: *Green compacts; sintering; bending strength; microstructure.*

1. Introduction

The applications of metal in industry were started since long time ago and there are many kind of metals have been utilized for developments according to their properties and characteristics. Although the performance of metal was great but for individual material of metal (pure element) such as iron, copper, aluminum and others had to face difficulty due to their disadvantages [1]. Iron was known due to their strength but when exposing to certain environments it will be highly corrosive material and for copper, soft characteristic has become disadvantage as well as aluminum for overpriced and not strong enough [2]. In order to mitigate this issue, alloying method was applied to mix two or more element either among metal or with non-metal (one must be metal) to form a coherent mass known as alloy [3]. The structure of alloy consists of the base metal (major constituent) and the alloying elements (metal or non-metal) commonly in a small quantity to form the product based on the desire properties. Nowadays, the most selective alloying technique by manufacturers is powder metallurgy instead of foundry process and mechanical alloying. Powder metallurgy is a forming process of solid components/parts by using powder elements through compaction and sintering. Powder metallurgy was selected due to the disadvantages of foundry process and mechanical alloying such as high thermal energy is required to melt the matrix and a lot of further processing steps to develop a final product since only alloy billets can be produced. Furthermore, powder metallurgy is able to provide low production, cutting production time by reducing the number of processing steps, no extra scrap losses, energy efficient technique and near-net-shape products [4].

In compaction, warm powder compaction was introduced to improve cold powder compaction by installing the heater on the die compaction rig. The performance of warm powder compaction has been proven by the density of green product has been increased drastically. The forming temperature can be manipulated in order to identify the optimum density of the green product before going to sintering process. High strength of green compacts is essential to ensure the sintered products to have higher density and strength since they are related to each other [5]. The friction occurs during compaction between the powders and die wall as well as among the powder particles. This is the most critical issue since friction has high potential to influence the density and the microstructure of the green compact especially during ejection stage. The effects of friction to the products are exposed to fracture mechanism, bad surface finishing and shortened the life span of die compaction rig [6-7]. The lubricant has been utilized in order to reduce the friction and ejection force as well. From the previous research studies, most of iron based alloys used zinc stearate as its lubricant due to compatibility among the powder particles and suitable for warm powder compaction since it was able to develop higher density products. Zinc stearate is admixed with the powder mass before compaction and the amount of lubricant must be sufficient. The range of lubricant percentage is around 0.25% to 0.7% according to the previous study [8]. If the excessive amount of lubricant is used in the powder mass, it results to reduce the density of the green compacts and subsequently increases the cost due to some additional amount of lubricant.

Furthermore, the strength of green compact with zinc stearate is higher compared to green compact without lubricant after compacting at different pressures [9-10].
Sintering is a process after compaction where the heat treatment is applied to a powder compact in order to gain and improve the strength by welded together the powder particles to form a coherent mass. The sintering temperature must be below the melting point of main constituent. The basic principle of sintering is the achieving rate of the desired degree of bonding among the particles in powder compacts. During sintering, densification and recrystallization occur through material transport mechanisms (surface diffusion, grain boundary diffusion, plastic flow, lattice diffusion, and vapor transport) and can be controlled by using sintering parameters (sintering temperature, sintering rate and holding time) [11-12]. The microstructure and porosity depend on the sintering parameters and represent as the degree of particles bonding. The combination of solid state sintering and liquid state sintering in the process embark the changes and rearrangement of grain particles since liquid state sintering accelerates the occupying of pores among the grains. Effect of the microstructure and porosity on the sintered product resulted in changes of relative density, electrical resistivity, flexure stress and microstructures. Therefore, the purpose of this paper is to investigate the effect of sintering temperature and holding time to the FeCuAl powder compacts formed through warm powder compaction route.

2. Experimental Procedure

The experimental works were conducted through four consecutive phases which are initially started with feedstock preparation, followed by defect-free green compact generation, sintering under controlled argon gas atmosphere and lastly, sample characterization. The feedstock was prepared by using iron powder ASC100.29 with 20-180 µm of particle size (91.7 wt%), copper (7.5 wt%), aluminum (0.5 wt%), and zinc stearate (0.3 wt%) as lubricant. The whole powders including main powder, elemental powder and lubricant were mixed mechanically at a rotation of 30 rpm for 30 min. The mixed powder was filled inside the die cavity at the T-15 compaction rig (Fig. 1) which was equipped with heating system. Solid cylindrical shape green compacts (Fig. 1) were generated by compacting the powder mass at 150˚C by applying 425 MPa of axial loading simultaneously from downward and upward directions.

Fig. 1: T-15 dies compaction rig

Fig. 2: Solid cylindrical shape FeCuAl green compact

The defect-free green compacts were then sintered by using a custom made argon gas fired furnace (Model: HT3-1400-SIC, S/N: LT007) at three different temperatures, i.e., 800˚C, 900˚C and 1000˚C for 120 min, 150 min and 180 min, respectively, under constant sintering rate of 10˚C/min. The ceramic tube was mounted inside the custom made sintering furnace with the dimensions, i.e., length was 150 mm, inner diameter was 40 mm and outer diameter was 50 mm. The sintered samples/final products were characterized for their physical properties, electrical properties, mechanical properties and their microstructures were evaluated. The dimensional measurement was performed by using a digital Vernier calliper (Brand: Mitutoyo, Model: CD-6”CS, S/N: 04171546, Accuracy: +/- 0.001 in). The mass and volume of the final product were measured in order to calculate the relative density. The electrical resistivity was measured by using a digital multimeter (Brand: Fluke, Model: Fluke 115, S/N: 28341190ws) and the flexure stress was measured by using three point bending test machine (Brand: Instron, Model: Instron 3365, S/N: SAA61569) following the standard (ASTM E290-09). The images of the fractured surface of each sample was captured through scanning electron microscopy (Brand: JEOL, Model: JSM-6010PLUS/LA).

3. Results and Discussion

The results of relative density of sintered samples are represented in Fig. 3. According to the results, the relative density has been influenced by the sintering temperature and holding time. The samples sintered at 800˚C and 900˚C obtained higher relative density compared to samples sintered at 1000˚C although at different holding time was applied. The highest relative density was achieved by the sample sintered at 800˚C with 150 min of holding time. The relative density was found to decrease when the holding time was increased. Longer holding time is not suitable for FeCuAl at high sintering temperature and it might be due to the transition to liquid state sintering. The presence of liquid state sintering is not suitable for high sintering temperature moreover when the gap between melting point of elemental powder (copper and aluminum) and sintering temperature are high. Liquid state sintering is only work at lower sintering temperature in order to lead capillary forces among particles that can result in densification. Furthermore, the factors which might be the reason why liquid state sintering is not suitable for FeCuAl to produce high relative density are due to high distortion and deterioration of mechanical properties because of the solidification of brittle phases along grain boundaries and/or grain growth during sintering [13].

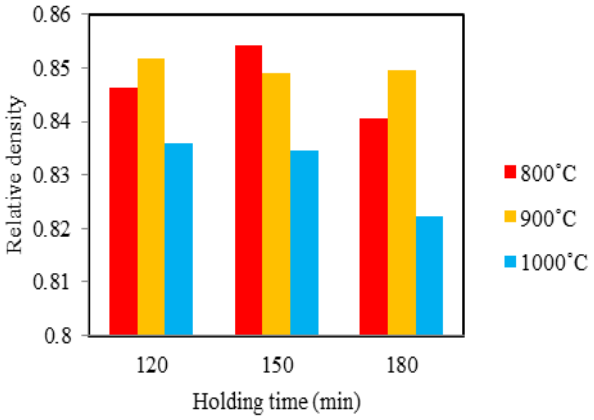


Fig. 3: Relative density of samples sintered for different holding time

The electrical resistivity results are shown in Fig. 4. All the sintered samples have higher electrical resistivity when the holding time is increased at all sintering temperature (800˚C, 900˚C and 1000˚C). The samples sintered at 900˚C have contributed to highest electrical resistivity at three different holding times. The samples have higher electrical resistivity might be due to the higher porosity and it was supported in the microstructure images in Fig. 7. Electrical resistivity is inversely proportional to the electrical conductivity where higher electrical resistivity prevents electrical current to pass through the samples.

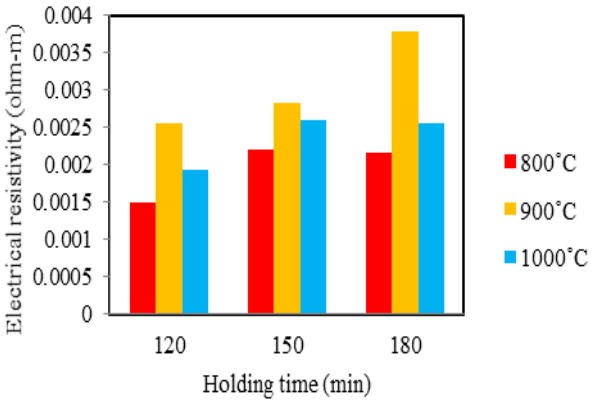


Fig. 4: Electrical resistivity of samples sintered for different holding time

The results of flexure stress are shown in Fig. 5. According to the results, the flexure stress is found to be influenced by sintering temperature and holding time. The samples sintered at 1000˚C have higher flexure stress at each holding time instead of samples sintered at 800˚C and 900˚C. Highest flexure stress was represented by the sample sintered at 1000˚C and 120 min of holding time. The flexure stress become higher due to the inter-particles bonding is higher compared to the number interconnected pores. The flexure stress tends to become lower when the holding time is increased for those three sintering temperatures. The connection between flexure stress and porosity are shown in the microstructure images as in Figs. 6, 7 and 8, the number of pores have started to grow and insisted to create the grains boundary which could initiate crack propagation inside the samples when holding time is increased at each sintering temperature. As in increasing of holding time, the structure of powder particles at 120 min of holding time at each sintering temperature tends to have uniform grains arrangement due to the necking process through the transport mechanisms which accelerated by liquid state sintering to fulfil the pores among the powder particles [14].

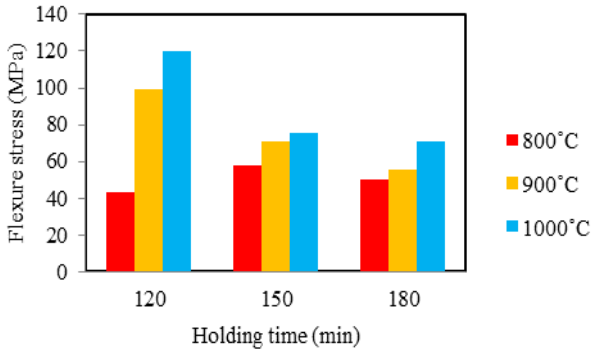
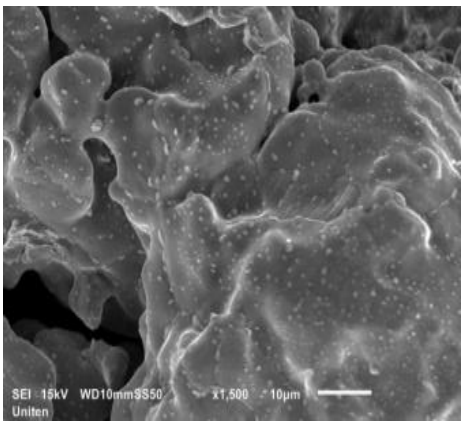


Fig. 5: Flexure stress of samples sintered for different holding time



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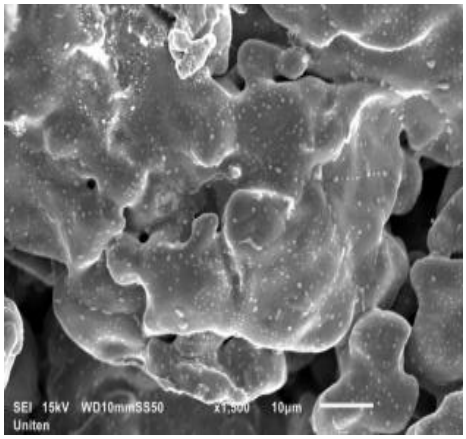

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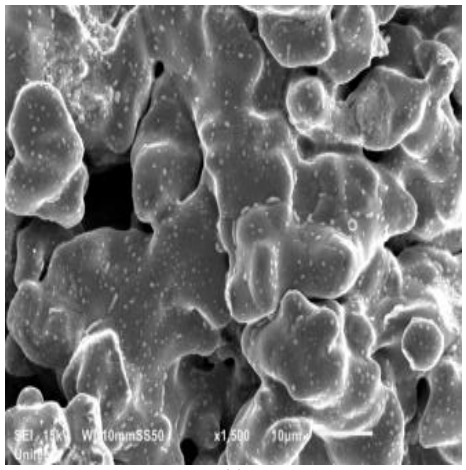
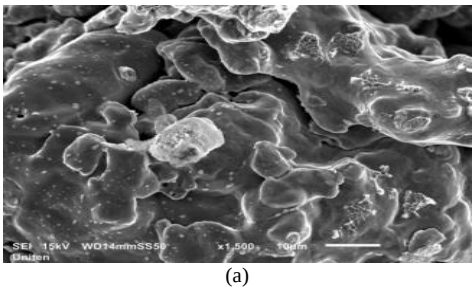


(c)

Fig. 6: Samples sintered at 1000˚C for three different holding times (a) 120 min, (b) 150 min (c) 180 min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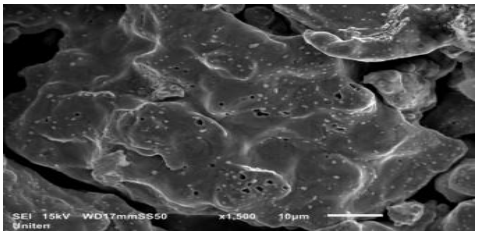
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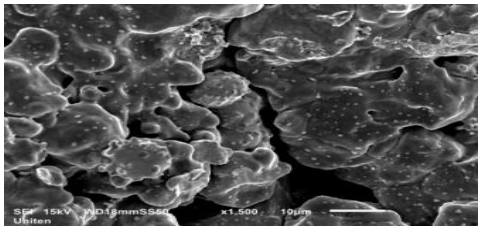
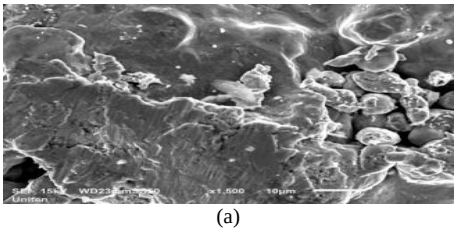

(c)

Fig. 7: Samples sintered at 900˚C for three different holding times (a) 120 min, (b) 150 min (c) 180 min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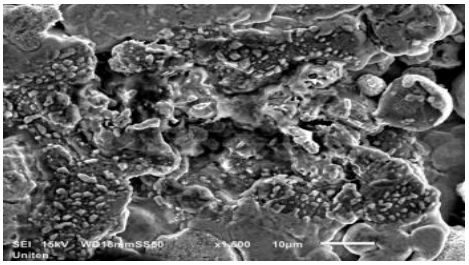
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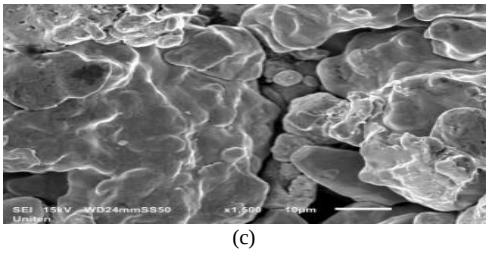
(c)

Fig. 8: Samples sintered at 800˚C for three different holding times (a) 120 min, (b) 150 min (c) 180 min

4. Conclusion

Sintering temperature and holding time are found to influence the properties of FeCuAl powder compacts formed at elevated temperature. The results revealed that a sintering temperature of 1000˚C is able to generate products with higher flexure stress, i.e., 120 MPa but with lower relative density. A sintering temperature of 800˚C was found to provide higher relative density with low electrical resistivity and better microstructure. The final products having higher flexure stress, low electrical resistivity, higher relative density and with more uniform microstructure were obtained by sintering for 120 min. Longer holding time during sintering caused the growing of interconnected pores and grain boundary which could initiate crack growth and weakened the strength of the final product.

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