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An Experimental Investigation on the Effects of Forming Temperature and Sintering Schedule to the Final Characteristics of $Fe_{92}Cu_{7.5}Al_{0.5}$ Powder Compacts

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Abstract

This paper presents the development of FeCuAl powder compacts through warm compaction process. A lab-scale uni-axial die compaction rig was designed and fabricated which enabled the powder forming at elevated temperature. Iron powder ASC 100.29 was mechanically mixed with other elemental powders, i.e., copper (Cu), and aluminium (Al) for 30 minutes at a rotation of 30 rpm. Green compacts were then generated by forming the prepared feedstock at 30ºC (room temperature), 150ºC, and 200ºC through simultaneous upward and downward axial loadings of 325 MPa. The defect-free green compacts were subsequently sintered in argon gas fired furnace at 800ºC for three different holding times, i.e., 30, 60, and 90 minutes at three different rates, i.e., 5, 10, and 15ºC/min. The final products were characterized for their physical, electrical, and mechanical properties and their microstructures were evaluated. The results revealed that the suitable forming temperature is 150ºC, holding time is 30 minute, and sintering rate is 10ºC/min.

Keywords*:* FeCuAl alloy, warm compaction, sintering, sample characterization

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1. Background

In the area of materials processing technologies, there are several types of approaches to create or design new materials and parts consist of casting, forging, stamping, powder metallurgy, metal injection moulding and machining. Nowadays, majority of industries choose powder metallurgy method because this method has been already recognized and used in worldwide with numerous of significant advantages compared to others. Powder metallurgy (P/M) has been called as material processing which is to produce new product of materials by diffusing various types of powder materials as feedstock through the compaction and sintering process [1].

The reason why powder metallurgy has been selected is due to its advantages such as it is not a lengthy process as other manufacturing method and simultaneously gives more advantages in term of time saving, more economically, no extra scrap losses, saving energy and suitable for large number of production [2]. In addition, Near Net Shape (NNS) of the products or components are very important too in manufacturing industries. In this case, powder metallurgy is given as top position compared to

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other manufacturing methods in final products or components with absolute Near Net Shape (NNS).

Iron is the main source of structural material in the world and widely used for many applications due to its properties as strong, tough and importantly it is cheap. Unfortunately, at the same time iron also has been attached with some disadvantages such as highly corrosive and problem with strength to weight ratio [3]. As for copper, it has excellent ductility with excellent electrical conductivity and at the same time it was malleable but costly and soft type [4]. For aluminium, this material is a low density metal and has good performance in corrosion resistance properties as well as ductile material but very expensive [3]. Many researchers have tried to explore by doing a lot of research in order to create new design of material for improvement and development.

Powder compaction process is the productions of bulk powders from any material by compacting into preforms or dies of a desired shape and density [4, 10]. The product after compacting process is well known as a green compact which has enough strength to undergo further processes [10]. Green strength is commonly from mechanical interlocking of irregularities on the particle surfaces due to plastic deformation during pressing [11]. Powder compaction process is divided into two types, first is powders are compacted at room or ambient temperature

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and second type is the compaction process performed at elevated temperature. Both types of compaction process have different result at the end of the studies [4].

In general understanding, sintering known as a process of powder particles bonding by molecular or atomic attraction to form a coherent body and at the same time under influence of heat application, making the strength of powder mass become higher and resulting some changes in densification and recrystallization by material transport [5]. The application of heat for this treatment is prepared below melting point of powder material which is around 60 - 80% of the melting temperature of the main powder constituent for a certain time in order to improve inter-particle bonding [6]. Sintering schedule is also considered as an important part in enhancement process similar like other factors such as green compact density, particle size, lubrication, geometrical structure and sintering atmosphere in order to achieve desired final result in term of strength and density [7].

In addition, sintering schedule is comprehended by the temperature, heating rate or cooling rate, and holding time. Temperature for sintering process plays an important role as it influences the interaction among particles based on the type of material used in the process. Generally, temperature is considered properly at the beginning of the experiment in order to decide whether to include solid state sintering only or with liquid state sintering into the process. On the other hand, there is a correlation between temperature and time, when sintering temperature is done at higher temperature then the time consumption for this process becomes be short [8].

During the sintering process, usually diffusion acts as an important role. Generally, diffusions are formed by surface diffusion, volume, or from grain boundary. Density is a significant factor which requested by many applications and it has been provided by several methods such as solid state sintering, liquid phase sintering or activated sintering. Once density is increased, simultaneously other properties such as fatigue, ductility, strength and modulus of elasticity are also improved [9].

In order to have high performance final product of FeCuAl by using powder compaction method, the proper forming and sintering mechanisms need to be identified with respected to each powder properties. So far, there is no published results found on the effect of forming temperature and sintering schedule to the characteristics of sintered products prepared through die compaction route. Therefore, the objective of this paper is to investigate the effects of forming temperature and sintering schedule to the sintered properties of FeCuAl powder compacts.

2. Experimental Procedure

In the implementation of experiment, there are four main consecutive steps need to be considered properly which consist of feedstock preparation, green sample generation, sintering and sample characterization. The main powder

constituent used for this experiment is iron powder ASC 100.29 with particle size range between 20 to 180 μm. The compositions of powder used are copper with 7.5% (less than 10 μ m), aluminium with 0.5% (200 mesh or 74 μ m) and the balance was iron powder. Each powder was combined together in order to mix mechanically for 30 minutes at 30 rpm by using Stuart rotator SB3 mixer. The mixed powder mass was filled into the rectangular die cavity and compacted at 30°C (room temperature), 150°C and 200°C by the 130 kN applied load from upper and lower punches, simultaneously.

The defect-free green compacts that generated from the compaction process were subsequently sintered by using custom made argon gas fired furnace at uniform sintering temperature, 800°C. The sintering furnace was customized in order to mount with the ceramic tube that has dimensions as the outer diameter is 50 mm, the inner diameter is 40 mm and the hot length 150 mm. The green compacts were sintered at three sintering rates for heating and cooling processes which are 5°C/min, 10°C/min and 15°C/min with sintering time of 30 minutes, 60 minutes and 90 minutes, respectively. The final products that generated from sintering process were characterized for their physical, mechanical, and electrical properties and their microstructures were evaluated through scanning electron microscope (SEM). The dimension, density, electrical resistivity, bending strength (three point bending machine) and microstructure were measured and evaluated for all the final products.

3. Results and Discussion

Relative densities of samples formed at room temperature (30°C) and elevated temperature (150°C and 200°C) with different sintering parameter at 5°C/min - 15°C/min for 30 minutes - 90 minutes are shown in Figs. 1 - 3. It indicated that the relative density of the sample at elevated temperature is higher compared to at room temperature. In addition, higher relative density means that the sample has lower number of pores inside it and compacted properly. The highest relative density obtained through this experiment is by the sample formed at 150°C at rate of 5°C/min with holding time 60 minutes. Generally, the effects of sintering schedule to relative density of samples are not consistent and unpredictable.

In term of volumetric expansion, all the samples have been enlarged after undergoing sintering process due to swelling based on the Figs. 4 - 6. Apart from that, swelling particularly would affect the relative density of the final product because density is inversely proportional to the volume. Eventually, the final products with lower volumetric expansion would have higher relative density where most of the samples are generated at 150°C. Effect of sintering schedule showed that the holding time and sintering rate provided some interaction to the particles inside the sample. Based on holding time result, 30 minutes has provided volumetric expansion at low position for all

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three different sintering rates at 150°C forming temperature.

Fig. 1: Relative density of samples formed at 30°C and sintered at different sintering rates and holding times

Fig. 2: Relative density of samples formed at 150°C and sintered at different sintering rates and holding times

Fig. 3: Relative density of samples formed at 200°C and sintered at different sintering rates and holding times

Fig. 4: Volumetric expansions (percentage) of samples formed at 30°C and sintered at different sintering rates and holding times

Fig. 5: Volumetric expansions (percentage) of samples formed at 150°C and sintered at different sintering rates and holding time

Fig. 6: Volumetric expansions (percentage) of samples formed at 200°C and sintered at different sintering rates and holding times

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Overall, all the samples have different value of electrical resistivity as shown in Figs. 7 - 9. Most of the samples that have higher electrical resistivity are formed at 200°C. Thus, this is might be due to effect of alloy forming among iron, copper and aluminium. Generally, alloying process can be one of the factors in increment of electrical resistivity especially when the sample has been already gone through high forming temperature which is 200°C. In this case, the effect of sintering schedule is inconsistent and the pattern is very hard to identify. Theoretically, higher electrical resistivity comes out with lower electrical conductivity which means the current flow that can go through the sample is low. On the other hand, samples that have higher density can pass more electrical current through them.

Fig. 7: Electrical resistivity of (compaction direction) sintered samples formed at 30°C

Fig. 8: Electrical resistivity of (compaction direction) sintered samples formed at 150°C

Fig. 9: Electrical resistivity of (compaction direction) sintered samples formed at 200°C

Through the obtained results in Figs. 11 and 12, samples which formed at elevated temperature or currently known as warm compaction process came out with higher strength compared to compaction at room temperature (Fig. 10). Generally, most of higher bending strength of final products are from 150°C forming temperature due to the material particles which are affected by the heat supplied during the compaction and also heat treatment by sintering using different parameters. The particles of the material started to react on each other as the transport mechanisms began in order to fill the leave pores. Theoretically, bending strength has a correlation with the pores which is inversely proportional to each other. As the bending strength or flexure stress increases, the number of pores becomes low.

According to the flexure stress results in Figs. 10 - 12, overall sintering rate did not show consistent results. Apart from that, most of the samples that have higher flexure stress are contributed by 10°C/min compared to 5°C/min and 15°C/min. It is observed that a sintering rate of 10°C/min is ideal for this process because if the rate is too low or high, it definitely would cause the rearrangement of the particles of the material and their interaction among particles became weaker due to too slow or fast reaction until it reached the room temperature.

The flexure stress results (Fig. 11) showed that 10°C/min sintering rate commonly reacts as a peak in flexure stress data compared to others and the highest flexure stress is also obtained at this sintering rate. Furthermore, there is a correlation between microstructure and flexure stress where higher flexure stress come out with a good microstructure arrangement as well as less interconnected pores and good inter-particles bonding. From the obtained results (Figs. 13 - 15), majority of the samples with higher flexure stress came out with a better microstructure. In addition, as microstructures are formed with less number of pores, its electrical resistivity value is decreased because of the fact that current can flow smoothly in the sample without any resistance.

Fig. 10: Flexure stress at 30°C forming temperature and sintered at different sintering rates and holding times

Fig. 11: Flexure stress at 150°C forming temperature and sintered at different sintering rates and holding times

Fig. 12: Flexure stress at 200°C forming temperature and sintered at different sintering rates and holding times

Fig. 13: Microstructures of sample formed at 150°C and sintered for 60 minutes at 5°C/min

Fig. 14: Microstructures of sample formed at 150°C and sintered for 60 minutes at 10°C/min

Fig. 15: Microstructures of sample formed at 150°C and sintered for 60 minutes at 15°C/min

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4. Conclusion

Based on the obtained results, the most suitable forming and sintering parameters for FeCuAl powder mixture are forming temperature is 150°C, sintered for 30 minutes at 10°C/min sintering rate at constant sintering temperature of 800°C. This parameters could led to create higher sintered density means more smaller and compact final products, low electrical resistivity means the current can flow at higher rate and can be used as electrical conductivity purpose. These parameters are also developed better microstructure as well as high bending strength in order to generate high ductility products or components which can be used in manufacturing industry and daily applications.

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