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Abstract. This paper presents the outcomes of an experimental investigation regarding the sintering characteristics of FeCuAl powder compacts formed through uniaxial die compaction process at elevated temperature. A lab-scale uniaxial die compaction rig was designed and fabricated in order to shape powder mass at elevated temperature. Iron powder ASC 100.29 (92 wt%) was blended mechanically together with other elemental powders, i.e., copper (7.5 wt%), and aluminum (0.5 wt%) at a rotation of 30 rpm for 30 min. The prepared feedstock was shaped at 200ºC through simultaneous upward and downward axial loadings of 325 MPa and the product generated through this process is known as green compacts. The defect-free green compacts were subsequently sintered in argon gas fired furnace at constant temperature, i.e., 800ºC at three different sintering rates, i.e., 5, 10 and 15ºC/min for three different holding times, i.e., 30, 60 and 90 min, respectively. The final products were then characterized for their physical, electrical, and mechanical properties and their microstructures were evaluated. The results revealed that the sample sintered for a shorter period of time, i.e., 30 min at a moderate heating/cooling rate, i.e., 10ºC/min obtained the better final characteristics, i.e., higher relative density, lower volumetric expansion, lower electrical resistivity, higher strength and more uniform microstructure.

1. Introduction

In the area of materials processing technology, there are several approaches to create or design new materials where powder metallurgy has been selected by most of the manufacturers since this method has been already recognized and used in worldwide with numerous significant advantages compared to others. Powder metallurgy is a process where a powder or powder mass is compacted by pressing it in order to form into a desired shape [1-2]. Powder metallurgy is divided into (i) isostatic pressing, (ii) injection moulding, and (iii) powder compaction. Isostatic pressing can be conducted either at room temperature, i.e., cold isostatic pressing or at elevated temperature, i.e., hot isostatic pressing. Powder compaction is also can be conducted at room temperature, i.e., cold compaction or at elevated temperature but below the recrystallization temperature of the main powder constituent called warm compaction [3-4].

There are many reasons why powder metallurgy has been selected in manufacturing industries compared to others such as (i) shorter production time, i.e., it does not require lengthy process compared to other manufacturing methods, (ii) cheaper production cost or more economical, (iii) no extra scrap losses, (iv) energy savings, and (v) suitable for large volume of production. Near-net-shape products or components are very important in manufacturing industries hence powder

metallurgy is given as the top position compared to other manufacturing methods in manufacturing final products or components with absolute near-net-shape [5-7].

The combination of iron, copper, and aluminum are selected to overcome their individual material weaknesses such as iron corrodes easily and faces strength to weight ratio problem, copper and aluminum are expensive and very soft type of materials. Composition of elemental powder is very important because it has major effect on properties. Each elemental powder has different properties and to combine with other types of powder, there are several significant factors need to be considered, i.e., their weaknesses, advantages, characteristics, quantities and qualities along with their applications in daily life [8].

Powder compaction is the production of any component from powder material by pressing it to form into desired shape. The product after compaction process is known as a green compact, which has enough strength to be handled to the next process. The strength of green compact, which is mechanical strength is known as green strength. Green strength is formed from interaction of mechanical interlocking that occurred on the surfaces of particle and at the same time, it is contributed by plastic deformation during process of pressing. Powder compaction can either be conducted at room temperature or at elevated temperature but below the recrystallization temperature of the main powder constituent. The first type of compaction is termed as cold compaction process whereas the second type is termed as warm compaction process. The warm compaction process was introduced in late nineties by realizing the fact that temperature rise softens the metal powder hence higher green density could be achieved if a powder mass is formed at above room temperature [9-10].

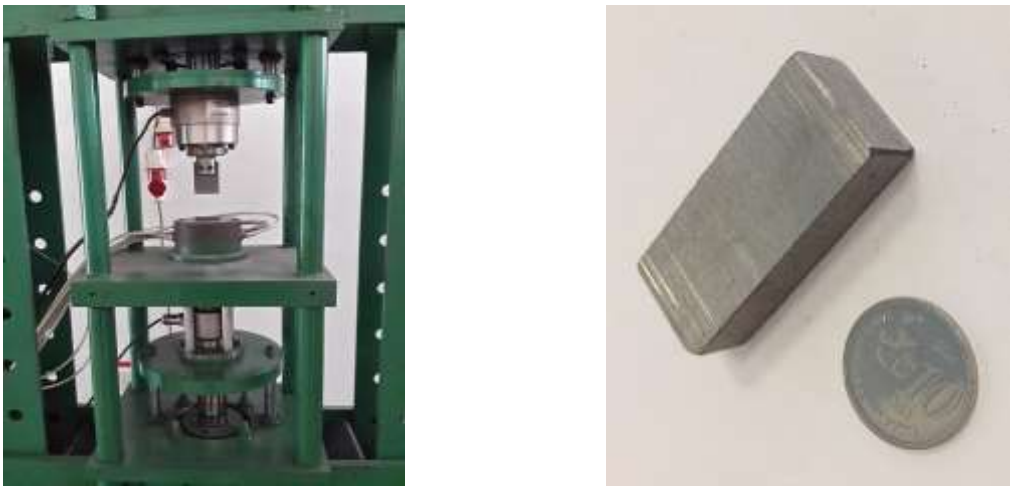
Sintering is the last step in a full cycle of powder compaction process, which is a thermally activated material transport in a powder compact in order to reduce surface contact among particles by growing process, shrinkage of pore volume and change of pore geometry. Sintering is known as a process of powder particles bonding by molecular or atomic attraction to form a coherent body and at the same time due to the heat application, the strength of the powder mass becomes higher and resulting some changes in densification and recrystallization by material transport. The application of heat for this treatment is set up below melting point of main powder constituent, which is around 60% - 80% for a certain duration of time in order to improve inter-particle bonding. Sintering is governed by several significant variables, i.e., temperature, time, and rate. These variables can be controlled in order to increase density of a product with better microstructure and less porosity [11-12].

Sintering can be divided into solid state sintering and liquid state sintering. Both types of sintering are possible to occur at the same time based on the melting point of individual material. Diffusion acts an important role during sintering, which is governed by surface diffusion, volume or grain boundary diffusion. Density is a significant factor for many applications, which can be improved through solid state sintering, liquid state sintering or combination of both. Products with increased density could simultaneously improve the other properties, i.e., fatigue, ductility, strength, and modulus of elasticity. The transport mechanisms that commonly involved in solid state sintering are volume diffusion, surface diffusion, grain-boundary diffusion, atoms evaporated on surfaces, and viscous or plastic flow. Liquid state sintering is related to formation of liquid phase during sintering due to lower melting point of element inside green compacts. Liquid state sintering is commonly practised to sinter alloys and composites that melt below temperature range used for sintering. Due to this solubility process, the attraction occurs among the grains during the wetting process of liquid to solid in order to unite the grains. Solid-liquid solubility relations between liquid material and solid material are very significant in this process in order to create coherent bonding among the particles [13-15].

A lot of research activities are reported on the warm metal powder compaction by using a single metallic material or by adding powder type lubricants or additives to enhance the characteristics of final products. Since sintering is a key step to manufacture new products or components through warm powder compaction method and for combination of iron (Fe), copper (Cu) and aluminium (Al), there is still a lot of research gaps need to be explored in order to find out the most suitable variables and moreover. Furthermore, FeCuAl is not commercially available in the market because it is a new kind of alloy. Therefore, the objective of this paper is to study the effects of sintering schedule to the final properties of FeCuAl powder mass formed at elevated temperature.

2. Materials and Methods

Four consecutive steps are involved in this experiment, i.e., (i) feedstock preparation, (ii) green compact generation, (iii) sintering in argon gas fired environment, and (iv) sample characterization. Iron powder ASC 100.29 with particle size range of 20 - 180 μm (92 wt%) was used as the main powder constituent whereas copper (7.5 wt%) and aluminum (0.5 wt%) were used as other elemental powders. The main powder and elemental powder were mixed mechanically for 30 min at a rotation of 30 rpm. The mixed powder mass was filled into the die cavity located at the warm compaction rig (Figure 1(a)) and rectangular shape green compacts (Figure 1(b) were generated by compacting the powder mass at 200ºC through simultaneous upward and downward axial loadings of 325 MPa.



(a) (b)

Figure 1. (a). T-15 compaction rig with heating system.
(b). FeCuAl green compact.

The defect-free green compacts were subsequently sintered in a custom made argon fired furnace (Model: HT3-1400-SIC, S/N: LT007) at a constant sintering temperature of 800ºC at three different sintering rates, i.e., 5, 10 and 15ºC/min for 30, 60 and 90 min, respectively. The sintering furnace was customized in order to ensure the ceramic tube can be mounted as it was 150 mm long, 50 mm OD, and 40 mm ID. The final products were characterized for their physical, electrical, and mechanical properties and their microstructures were evaluated. The relative density of the products was calculated from the dimension and mass of the final products. The dimensional measurement was conducted by using a digital Vernier calliper (Brand: Mitutoyo, Model: CD-6”CS, S/N: 04171546, Accuracy: +/- 0.001 in). The electrical resistivity was measured by using a digital multimeter (Brand: Fluke, Model: Fluke 115, S/N: 28341190ws) whereas the flexure stress was measured through three point bending test machine (Brand: Instron, Model: Instron 3365, S/N: SAA61569) following the standard (ASTM E290-09). The image of the fractured surface of each sample was captured through scanning electron microscopy (Brand: JEOL, Model: JSM- 6010PLUS/LA).

3. Results and Discussion

Relative densities of samples sintered at different schedules are depicted in Figure 2. It can clearly be observed in Figure 2 that the highest relative density was obtained by the sample sintered for 90 min at a rate of 5ºC/min whereas the lowest relative density was obtained by the sample sintered for 30 min at the same rate. Sintering for longer time permits the perfect necking of particles hence the size of the product becomes smaller. Neck growth also strengthens the product.

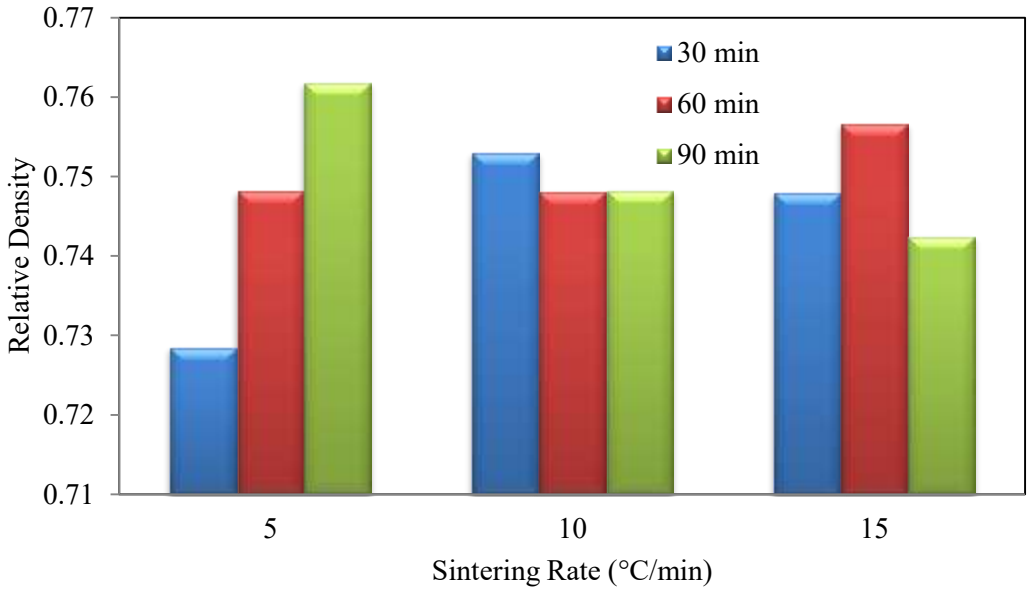


Figure 2. Relative density of sintered products.

Figure 3 shows the dimensional change of the samples sintered at different schedule. It is clear from the figure that every sample experienced swelling during sintering. The highest swelling was occurred at the sample sintered for 30 min at a rate of 10ºC/min whereas the lowest volumetric expansion was occurred at the sample sintered for 90 min at a rate of 5ºC/min. Swelling during sintering occurs mainly due to some factors, i.e., size of the particles, elemental composition, density of green compact, sintering temperature, and heating rate during the sintering processes. The results are also in line with relative density, i.e., higher volumetric expansion during sintering means lower relative density after sintering.

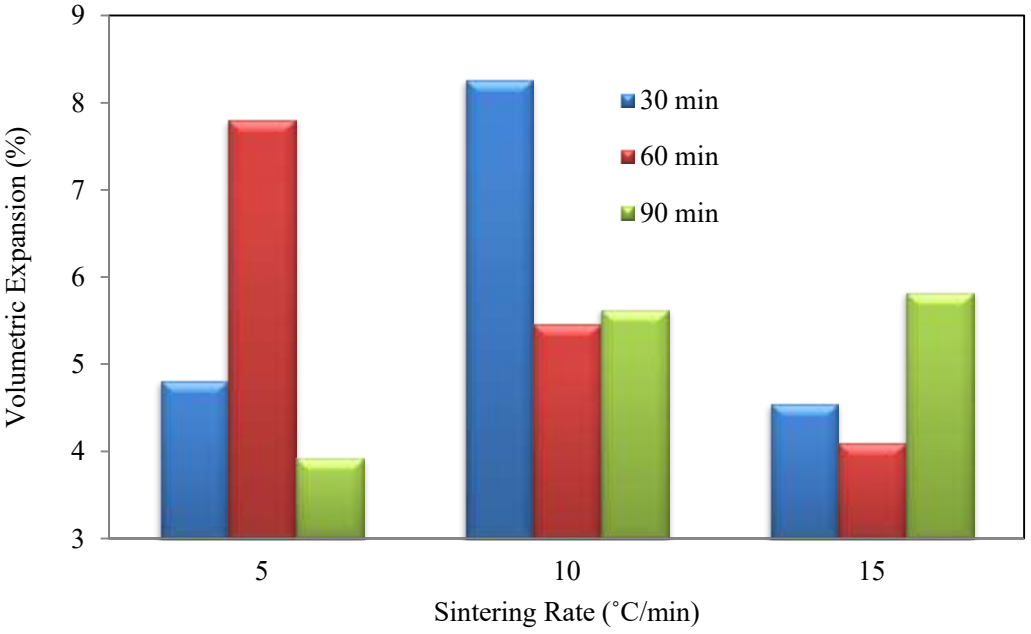


Figure 3. Volumetric expansion (percentage) of samples during sintering.

The electrical resistivity of the samples sintered at different schedule is depicted in Figure 4. Overall, the electrical resistivity values are found to be different at each sintering schedule. Electrical resistivity is the ability of a material to conduct electrical current. Since all the samples have the same composition, the electrical resistivity values were used as indicator of the integrity of the sample. Lower electrical resistivity value means the sample is able to flow electrical current easily means the possibility of the existence of interconnected pores or cracks inside the samples are smaller. Generally, the electrical resistivity was found to be higher at the samples sintered for longer period of time. Electrical resistivity was also found to be higher at the samples sintered at a higher sintering rate. Prolong exposure of the sample at argon gas fired environment might cause the formation of micro-cracks inside the sample which might lower down the electrical conductivity means increased the electrical resistivity.

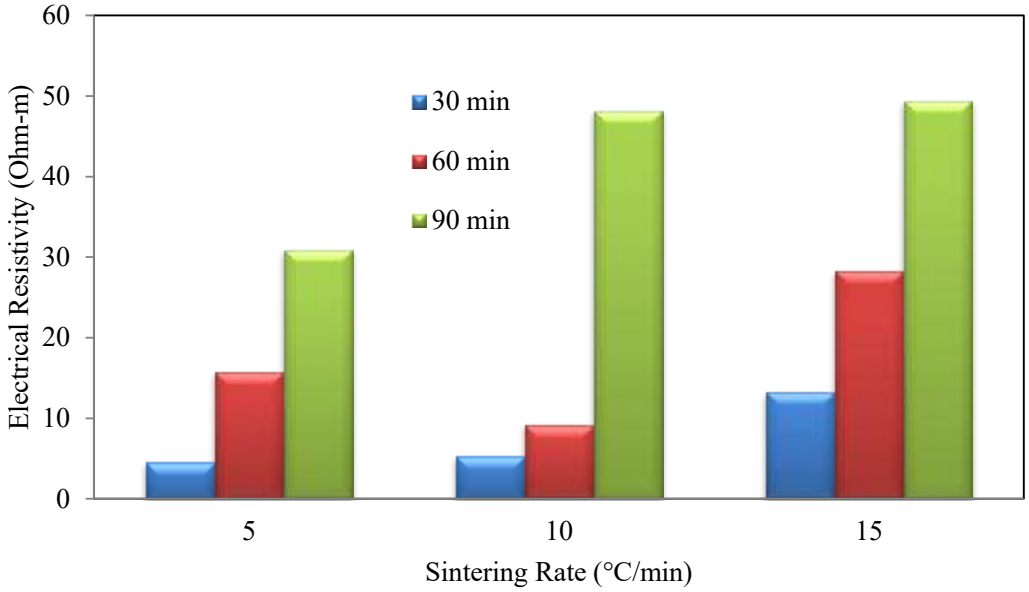


Figure 4. Electrical resistivity of the sintered products.

Flexure stress or three point bending strength of the sintered samples are presented in Figure 5. It is clear that higher bending strength was obtained by the sample sintered for 90 min at rate of 5ºC/min. Generally, the material particles are affected during the sintering process. The particles of the material started to react on each other as the transport mechanism begins in order to fill the pores. Since the melting point of aluminum is less than the sintering temperature, liquid state sintering by aluminum helped to improve inter-particle bonding among particles as molten aluminum would fill the pores and functioned to attract iron and copper particles to close the gaps/pores among them. Effectiveness of sintering schedule to the flexure stress has been identified where most of the samples that have lower flexure stress were sintered for 30 min compared to others. Therefore, a holding time of 30 min is found to be not enough to particles movement and rearrangement of the grains to develop higher strength of the final products.

The SEM images of fractured surface of samples sintered at different schedule are shown in Figures 6-8. The microstructures are found to be in line with the strength where the sintered samples of higher flexure stress have good inter-particle bonding and low interconnected pores. The microstructure of samples sintered at a rate of 10°C/min shown uniform grain rearrangement compared to the samples sintered at 5°C/min and 15°C/min.

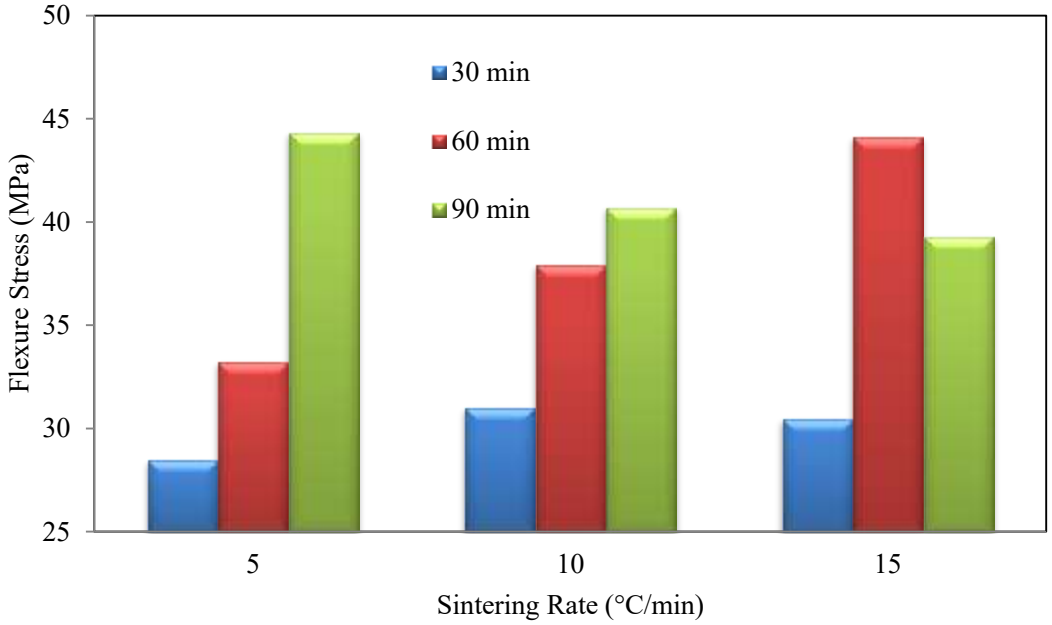
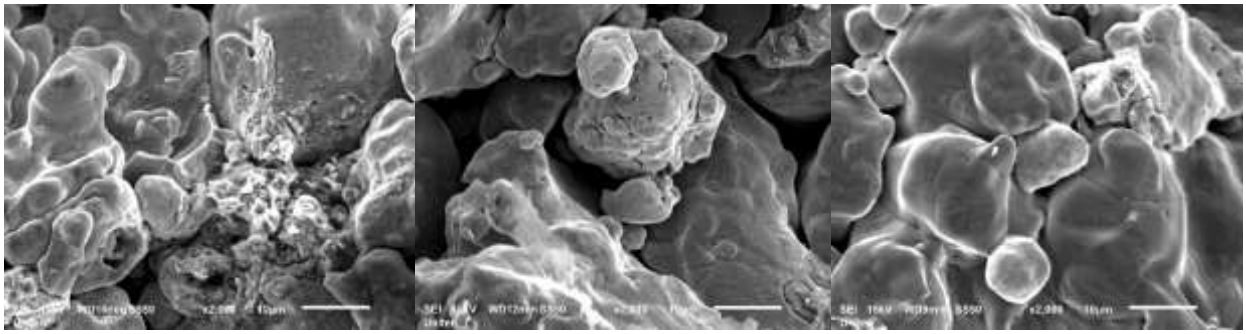
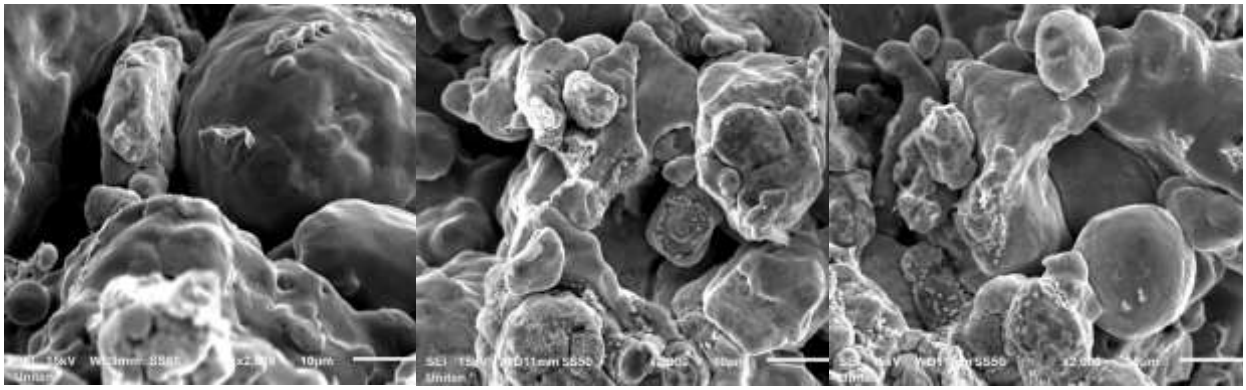


Figure 5. Flexure stress of sintered products.



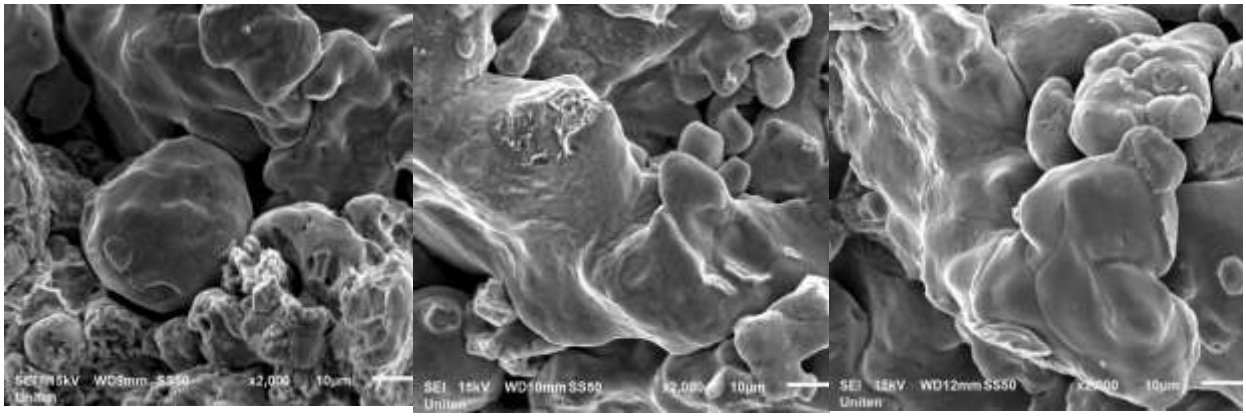
30 min 60 min 90 min

Figure 6. Microstructure of products sintered at a rate of 5ºC/min.



30 min 60 min 90 min

Figure 7. Microstructure of samples sintered at a rate of 10ºC/min.



30 min 60 min 90 min

Figure 8. Microstructure of samples sintered at a rate of 15ºC/min.

4. Conclusions

Sintering schedule was found to give impacts to the final properties of the products. Higher density final products of FeCuAl experienced lower volumetric expansion when sintered for 90 min at slow rate, i.e., 5ºC/min. However, prolong sintering time caused the formation of micro cracks inside the products, which caused the lower electrical conductivity. However, prolong sintering time also caused the proper necking among particles, which is manifested by the high flexure stress. Sintered products having more uniform microstructure were obtained by sintering the green compacts for 90 min at a slow heating/cooling rate, i.e., 5ºC/min.

Acknowledgements

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