Effects of forming temperature and sintering rate to the final properties of FeCuAl powder compacts formed through uniaxial die compaction process

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Abstract. This paper presents the outcomes of an experimental investigation on the effects of forming temperature and sintering schedule to the final characteristics of FeCuAl powder mass formed at different temperature and sintered at different schedule. A lab-scale uni-axial die compaction rig was designed and fabricated which enabled the compaction of powder mass at room temperature as well as elevated temperature. Iron (Fe) powder ASC 100.29 was mechanically mixed with other elemental powders, namely copper (Cu), and aluminum (Al) for 60 minutes and compacted at three different temperature, i.e., 30°C, 150°C, and 200°C by applying 425 MPa of simultaneous downward and upward axial loading to generate green compacts. The as-pressed samples were inspected visually and the defect-free green compacts were subsequently sintered in an argon gas fired furnace at 800°C for 60 min at three different heating/cooling rates, i.e., 5, 10, and 15°C/min, respectively. The sintered samples were then characterised for their physical, electrical, and mechanical properties. The microstructures of the sintered samples were also analysed. The results revealed that a forming temperature of 150°C and a sintering rate of 10°C/min could produce a product with better characteristics.

1. Introduction

Pure materials are proven to have a lot of disadvantages, i.e., soft, brittle, easy to degrade in hot and humid environment, etc. In order to overcome the disadvantages of pure materials, other materials are used as additives to produce a coherent mass named as alloy which is a homogeneous mixture of two or more materials where the dominant one is called matrix and the other materials are dissolved within it. Alloys have superior properties compared to individual pure materials [1-2]. Alloys are produced either through casting process or mechanical alloying. Casting process requires huge amount of thermal energy to melt the metals and subsequently machining operation to produce end products. On the other hand, mechanical alloying is used to prepare a solid solution of more than one metallic or non-metallic materials and subsequently the solid solution is pressed and sintered to produce end products. The solid solution is prepared through high energy ball milling of the intended powders for a long time. Both methods, i.e., casting and mechanical alloying are found to be time consuming and energy ineffective.
However, alloys could also be produced by shaping a mixture of intended powders and subsequently sintering the powder mass in inert gas fired furnace. This technique is relatively new but offers an approach to produce high quality end products [3]. However, no thorough study found in the literatures regarding this method of product manufacturing from of a mixture of materials. This method is considered as an alternative lower cost process to machining, casting, stamping, forging and other similar metal working technologies. Sintering is a part of a full cycle of powder compaction process, which must be done at controlled environment to avoid contamination of the component by other materials. The basic principle of sintering is the achieving rate of the desired degree of bonding among the particles in powder compacts. The sintering rate plays an important role in controlling the microstructure and porosity that determine the degree of particles bonding. The densification and the strengthening of particles inside the metal powder compacts reflect the properties of the sintered component. The sintered density relates directly to the dimensional and mass changes, which give perfect understanding of the densification process. Most importantly, the relative density of sintered parts determines the porosity in the sintered products. The sintered strength implies on the degree of particles bonding and particles arrangement at the surface of the compact, respectively. Therefore, the objective of this paper is to present the investigation of the effects of compaction temperature and sintering rate to the final properties of FeCuAl powder mass formed through uniaxial die compaction method.

2. Materials and Methods
The study consists of four consecutive steps, i.e, (i) feedstock preparation, (ii) green compact generation by powder forming through uniaxial die compaction method, (iii) pressureless sintering, and (iv) sintered sample characterization. Iron (Fe) powder ASC 100.29 of 20-180 µm particle size range was used as main powder constituent. The composition of copper (Cu) was 7.5 wt%, aluminum (Al) was 0.5% whereas 0.1 wt% of zinc stearate (Zn(C18H35O2)2) was added as inter-particle lubricant, and 0.1 wt% of fine carbon powder was used as additive. The main powder constituent and the other elemental powders were mixed mechanically for 60 minutes. The prepared feedstock was filled into the rectangular shape die cavity, then all the die assembled together with the powder mass was heated up to the desired temperature and kept for 30 minutes for uniform heating of the powder mass and the die assembly. The green samples were generated by compacting the powder mass at three different temperature, i.e., 30°C, 150°C, and 200°C through simultaneous upward and downward axial loadings of 425 MPa.

The green compacts were inspected visually and the defect-free ones were subsequently sintered using custom made argon gas fired sintering furnace (Model: HT3-1400-SIC, S/N: LT007). All the samples were sintered at 800°C, at three different sintering rates, i.e., 5°C/min, 10°C/min, and 15°C/min for a holding time of 60 min. Sintered samples were characterised for their physical, electrical, and mechanical properties as well as their microstructure was evaluated. The density of the products were 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) 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 electrical resistivity was measured by a digital multi-meter (Brand: Fluke, Model: Fluke 115, S/N: 28341190ws). 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
The relative density of final products compacted at different temperatures and sintered at different rates are depicted in figure 1 whereas the volumetric expansion of the same samples during sintering are presented in figure 2. It is clear from figure 1 that samples compacted at 30°C obtained the lowest relative density regardless of sintering rate. The samples compacted at 150°C were found to obtain the highest relative density. The relative density was found to decrease at the sample compacted at 200°C.
These phenomena are in line with volumetric expansion (figure 2) since the density is the ratio between the mass and the volume of the sample. Higher volumetric expansion caused the relative density of the samples to be lower. Deformation of metal powder mass at elevated temperature can be considered as a thermally activated process [5]. The powder particles soften and slide to fill the inter-particle voids hence the powder mass densifies easily compared to the compaction at room temperature [6]. The higher density obtained by the samples formed at 150°C might also be due to the use of zinc stearate as lubricant. Since the compaction temperature, i.e., 150°C was above the melting temperature of zinc stearate, i.e., 120-130°C, the zinc stearate melted during compaction, which probably reduced the friction among the powder particles [7]. Attainment of lower density for the compaction at 200°C is believed to be due to the thermal expansion of powder particles, which might cause the higher residual stress in green compacts hence volumetric expansion became bigger.

Figure 1. Relative density of sintered sample

Figure 2. Volumetric expansion of sintered sample

It is also interesting to highlight that the highest density is obtained by the samples sintered at slow rate, i.e., 5°C. Slow sintering rate caused the powder particles to form perfect necking among them which is manifested by smaller swelling [8] hence density did not drop much, i.e., less volumetric expansion (figure 2).

Electrical resistivity and bending strength are presented in figures 3 and 4. Highest electrical resistivity is found to be obtained by the sample formed at 200°C and sintered at a rate of 5°C. Higher electrical resistivity means lower electrical conductivity means it is difficult for the product to pass electrical current. Higher electrical resistivity is mainly due to the presence of interconnected pores or cracks inside the product.
Microstructures of sintered products are depicted in figure 5 where the microstructure of sample formed at 150°C and sintered at a rate of 10°C/min was found to be better (figure 5(b)) compared to the other products formed at different temperature and sintered at different rate. Less presence of grain growth is observed at that sample, which is in line with the strength of the sample. Slow sintering rate may cause the grain growth in metal powder mass during sintering which lessens load bearing capability of the products. Faster sintering rate also causes the initiation of micro-cracks inside the products due to the thermal shock hence lower down the strength of the products [9].

![Figure 3. Electrical resistivity of sintered sample](image1.png)  ![Figure 4. Bending strength of sintered sample](image2.png)

Figure 3. Electrical resistivity of sintered sample

Figure 4. Bending strength of sintered sample

4. Conclusion
The effects of forming temperature and sintering rate in producing bar shaped products from FeCuAl powder mass was investigated. The results revealed that the suitable forming temperature was 150°C and the suitable sintering rate was 10°C/min, which produced sintered product with higher relative density, lower volumetric expansion and electrical resistivity, higher bending strength and better microstructure.

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