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Effects of sintering time and temperature to the characteristics of FeCrAl powder compacts formed at elevated temperature

M M Rahman¹, H Y Rahman¹, M A A Awang¹ and I Sopyan²
¹Department of Mechanical Engineering, Universiti Tenaga Nasional, Putrajaya Campus, Jalan IKRAM-UNITEN, 43000 Kajang, Selangor, Malaysia
²Department of Manufacturing and Materials Engineering, Kulliyyah of Engineering, International Islamic University Malaysia, PO Box 10, 50728 Kuala Lumpur, Malaysia

Email:mujibur@uniten.edu.my

Abstract. This paper presents the outcomes of an experimental investigation on the effect of sintering schedule, i.e., holding time and temperature to the final properties of FeCrAl powder compacts prepared through uniaxial die compaction process at above room temperature. The feedstock was prepared by mechanically mixing iron powder ASC 100.29 with chromium (22 wt%) and aluminium (11 wt%) for 30 min at room temperature. A cylindrical shape die was filled with the powder mass and heated for one hour for uniform heating of the die assembly together with the powder mass. Once the temperature reached to the setup temperature, i.e., 150°C, the powder mass was formed by applying an axial pressure of 425 MPa simultaneously from upward and downward directions. The as-pressed green compacts were then cooled to room temperature and subsequently sintered in argon gas fired furnace at a rate of 5°C/min for three different holding times, i.e., 30, 60, and 90 min at three different sintering temperatures, i.e., 800, 900, and 1000°C. The sintered samples were characterized for their density, electrical resistivity, bending strength, and microstructure. The results revealed that the sample sintered at 1000°C for 90 min achieved the better characteristics.

1. Introduction
Pure metals are typically weak and soft at high temperature due to their malleability and ductility. By imparting other elements into a pure metal, a coherent mixture termed as alloy can be formed to enhance its properties [1-3]. FeCrAl alloy is used as engineering materials due to its high strength, improved form stability at high temperature, corrosion oxidation/resistance, and longer high temperature life. This alloy is formed through foundry or mechanical alloying process. Foundry process requires huge amount of heat to melt all the metals whereas mechanical alloying method requires the elements in powder form to be mixed by a high energy ball mill for a long time. Both methods require further processing to produce end products, which are time and energy consuming hence expensive. Another possibility is powder compaction process, which offers an approach to produce end products with minimal processing steps. However, research activities are lacking on the method of alloy forming through this route.

Sintering is a heat treatment process for bonding particles together into a coherent, predominantly solid structure via mass transport events. Such bonding improves the strength and other engineering
properties of powder compacts. The standard way of performing the sintering operation is to subject the parts to a high temperature in a controlled atmosphere using a sintering furnace. The process is known as free sintering since no mechanical stress is applied on the parts. The effect of sintering is the change of inter-particle contact, which begins with rapid growth among particles because of the cold welding taken place in the green compact. At this stage, the void volume is reduced and finally as the surface tension and diffusion continue, the spheroïdization of isolated pores occurs and it results in a relatively homogenous component. Correct sintering is of paramount importance to the powder metallurgy process to ensure not only the development of the strength needed for the part to fulfil its intended role as an engineering component, but also that the dimensions of the part are correct.

2. Materials and Methods
The experiments consist of four consecutive steps, i.e., (i) feedstock preparation, (ii) green sample generation, (iii) sintering, and (iv) product characterization. Iron powder ASC 100.29 having particle size range of 20 -180 μm was used as main powder constituent since this type of powder has been in use in most of the powder compaction industries [4]. Two other elemental powders, i.e., chromium (Cr) and aluminum (Al) were used as alloying element. The composition of iron powder was 67 (wt%) whereas the chromium was 22 (wt%) and the rest was aluminium powder. All of these elemental powders were mixed mechanically through a low speed (30 rpm) powder mixer for 30 min at room temperature [5]. A lab-scale uniaxial die compaction rig was designed and fabricated to shape metal powder mass at above ambient temperature. The cylindrical shape die cavity of 20 mm diameter was filled with the as-prepared feedstock. The top punch was brought closer to the upper level of the powder mass. The die assembly together with the powder mass was heated to 150°C and kept for 30 min for the uniform heating of the die assembly and the filled powder mass.

The powder mass was subsequently compacted by applying simultaneous downward and upward loading of 425 MPa. Once the desired loading is achieved, the top punch was kept at that position for a while for the settlement of the powder particles and brought back to the initial position which enabled the spring-back or release of residual stress from the powder compact [6]. The as-pressed powder compact termed as green compact was subsequently ejected from the die cavity by means of bottom punch. All the green samples were inspected visually for any defects or cracks. The defect-free samples were then sintered in a custom made argon gas fired furnace (Model: HT3-1400-SIC, S/N: LT007) at a rate of 5°C/min for three different holding times, i.e., 30, 60, and 90 min at three different sintering temperatures, i.e., 800, 900, and 1000°C.

Dimensions of the green as well as the sintered samples were measured to analyse the shape distortion from where the densities (green and sintered) were calculated. The dimensional measurement was conducted through a digital Vernier calliper (Model: CD-6"CS, S/N: 04171546) with an accuracy of +/- 0.01in. Electrical resistivity of the green as well as sintered samples were measured through a digital multimeter (Model: Fluke 115, S/N: 28341190ws). The purpose of electrical resistivity measurement was to detect the presence of any interconnected pores or cracks. 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). Each sample was hold tightly at both ends, force was applied at the middle of the sample until it was broken down. 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 difference in density from green to sintered product is presented in figure 1. The highest density drop was found at the sample sintered at 1000°C for 30 min, which is 18.19%. It can clearly be observed in figure 1 that the density difference increased when the sample was sintered at high temperature. All the samples sintered at 1000°C experienced higher volumetric expansion compared to the samples sintered at 900°C and 800°C for the sample holding time. Sintering of powder compacts at high temperature might cause the grain growth, which caused the swelling of compacts.
after sintering [7-8]. Another possible reason is the addition of aluminum, which has a lower melting temperature (667°C) compared to the other elements, i.e., iron and chromium. Mixed sintering might occur, which caused the significant swelling of the compacts after sintering.

Electrical resistivity difference from green to sintered is presented in figure 2 from where it is clear that samples sintered at 800°C and 1000°C experienced the highest electrical resistivity difference. Higher electrical resistivity means electrical current cannot flow easily through the samples. Higher electrical resistivity difference means micro-cracks or interconnected pores might be formed during sintering.

![Figure 1. Density difference after sintering.](image1)

![Figure 2. Electrical difference after sintering.](image2)

Bending strength or flexure stress of sintered samples is presented in figure 3. It can clearly be observed that samples sintered at 1000°C for 90 min obtained the highest bending strength. This finding is inline with electrical resistivity change (figure 2) where the sample sintered at 1000°C for 90 min experienced the negligible electrical resistivity change. This phenomenon might be due to the longer sintering time and higher sintering temperature caused the perfect sintering [9-10] of the samples. The microstructures of the samples sintered at 800°C, 900°C, and 1000°C for 90 min are shown in figures 4-6. It is clear from these figures (figures 4-6) that sample sintered at 90 contains less interconnected pores compared to the other samples.

![Figure 3. Bending strength of sintered samples.](image3)

![Figure 4. SEM image of sample sintered at 800°C for 90 min.](image4)
4. Conclusion
The effects of sintering temperature and holding time in producing solid cylindrical shape products from FeCrAl powder mass was investigated. The results revealed that the suitable sintering temperature was 1000ºC and the holding time was 90 min, which produced stronger sintered product with better microstructure.

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