Effect of Sintering Temperature and Holding Time to the Final Characteristics of Fecral Powder Compacts Formed at Elevated Temperature Through Die Compaction Method

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Abstract. This paper presents an experimental study on the effect of sintering temperature and holding time to the final characteristics of FeCrAl powder compacts formed at 200ºC through uniaxial die compaction process. The powder mass was prepared by mechanically mixing iron powder ASC 100.29 with chromium (22 wt%) and aluminum (11 wt%) for 30 min at room temperature. The powder mass was formed at 200ºC by applying an axial load of 425 MPa. The as-pressed compacts termed as green products were then cooled to room temperature and subsequently sintered in argon gas fired furnace at a rate of 5ºC/min for three different holding times, i.e., 30, 60, and 90 min at three different sintering temperatures, i.e., 800, 900, and 1000ºC. The sintered samples were characterized for their density, bending strength, and microstructure.

1. Introduction

A solid solution of two or more materials is termed as alloy provided that at least one of the materials is a metal. Matrix is the most dominant element of an alloy whereas the other elements are dissolved in it [1-3]. Since every single element has its own weakness which might not fulfil the design requirements, alloy is formed to overcome the weaknesses of individual materials. The main intention of making alloy is to improve the physical, chemical, mechanical, thermal, and electrical properties of materials, which can fulfill the application requirements [4-6]. Alloys are able to withstand structural integrity, surface, and property stability at elevated temperature under high mechanical stress and severe corrosive environment.

The most dominant element or matrix of FeCrAl alloy or usually termed as Fecralloy is the iron (Fe) which has a lot of disadvantages where the most critical ones are the strength degradation and corrosion at high temperature. When iron is mixed with chromium (Cr), a protective layer is formed which resists the corrosion. Since aluminum (Al) is soft and malleable, it enhances the formability [7].

The forming of mechanical components by compacting a single powder or a blend of more than one types of powder in a rigid container to a desired shape is defined as die compaction process [8]. The as-pressed powder mass or green products must have sufficient high density and strength to be handled for further treatment [9]. The advantages of forming of powder mass a above ambient temperature have
been identified by previous researchers [10-11], however the compaction must be conducted below the recrystallization temperature of the main powder constituent [12].

Sintering is an essential step of a full cycle of powder compaction process, which must be conducted at controlled environment to avoid contamination of the component by other materials [13]. The basic principle of sintering is the achieving rate of the desired degree of bonding among the particles in powder compacts [14]. The heating and cooling rate, sintering temperature, and holding time play important roles in controlling the microstructure and porosity that determine the degree of particles bonding. In solid state sintering, the bonding among particles requires material transport by volume diffusion (migration of vacancies), grain-boundary diffusion, surface diffusion, viscous or plastic flow, and evaporation/condensation of atoms on the surfaces. 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 [15].

It is clear from the explanation above that sintering schedule plays an important role in determining the quality of a final product where the green products are formed through powder compaction route. Current practice of FeCrAl alloy forming is through foundry process which is time consuming and energy inefficient hence expensive. There is also less thorough study found on the forming of FeCrAl alloy through powder compaction route. Therefore, the objective of this paper is to present the effect of sintering parameters, i.e., temperature and holding time to the final characteristics of FeCrAl powder compacts formed at above ambient temperature through die compaction route.

2. Materials and Methods

Iron powder ASC 100.29 manufactured by Höganäs AB with the composition of 1.5 wt% Cu, 0.5 wt% Mo, and 4 wt% Ni balanced with Fe [16] was used as main powder constituent. Chromium (Cr) powder (22 wt%) and aluminum (Al) powder (11 wt%) were used as alloying elements where the balance was Fe powder. The main powder constituent together with the other alloying elements were mixed mechanically for 30 min at room temperature at a rotation of 30 rpm.

The blended powder mass was formed through uniaxial die compaction process at a temperature of 200ºC by applying simultaneous downward and upward loading of 425 MPa. The as-pressed powder compact termed as green compact (Figure 1) was subsequently ejected from the die cavity by pushing the bottom punch to upward direction. 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 5ºC/min for three different holding times, i.e., 30, 60, and 90 min at three different sintering temperatures, i.e., 800, 900, and 1000ºC.

The green as well as the sintered densities of the samples were calculated from the mass and volume of each sample. The relative density was calculated by dividing the density of the individual product by the solid density. 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 (Figure 2). 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 densities of products sintered at different temperature for different times are depicted in Figures 3-4. It can clearly be observed that the relative density decreases as the sintering temperature increases irrespective of holding time. Longer holding time also caused the relative density to decrease. This finding is also in-line with volumetric expansion (Figure 5) where sintering at high temperature for a longer period of time caused the samples to swell. Since density was calculated from the mass of sintered product divided by its volume, therefore swelling of sintered product ended up with lower density. The swelling is believed to be due to the competition between classical sintering phenomena induces densification due to necking among powder particles (Figure 6) and material creeping under stresses caused by internal gas pressure [17]. Neck formation is driven by the surface diffusion which is usually the dominant mass transport mechanism during the early stage of sintering. The internal gas includes air trapped during powder forming and oxygen released from particle surface inside the pores that closed during compaction or sintering. Material creeping is believed to be the dominant mechanism at this current sintering temperature and holding time.

![Figure 1. FeCrAl green samples formed at 200°C](image1)

![Figure 2. Bending strength measurement (ASTM E290-09)](image2)

**Figure 3.** Relative density of the products sintered for different time

**Figure 4.** Relative density of the products sintered at different temperature
Flexure stress or bending strength of sintered samples is shown in Figure 7. Sample sintered at 1000°C for 30 min is found to obtain the highest strength. On the other hand, sample sintered at 800°C for 90 min obtained the lowest strength. The strength difference is also significant, i.e., strength of the products sintered at 1000°C is found to be almost double of the strength of the products sintered at 800°C and 900°C. It is believed that the samples were not fully sintered at temperatures 800°C and 900°C. Since the main powder constituent was the iron powder ASC100.29 which has the melting temperature of 1536°C. Therefore, sintering at a temperature lower 60% of the melting temperature of the main powder constituent does not allow the formation of necking which strengthen the sintered products [18].

Figures 8-10 show the microstructures of the products sintered at 800°C, 900°C and 1000°C for 30 min at a rate of 5°C/min. Bigger size and irregular shape pores are observed at the sample sintered at 800°C (Figure 8) as the pore size became smaller with increasing sintering temperature to 900°C (Figure 9) and 1000°C (Figure 10). Spherical shape pores are observed at the samples sintered at 900°C and 1000°C, however interconnected and irregular shape pores are found at sample sintered at 800°C. Interconnected pores weakened the sample (Figure 7) hence less force was required to break down the sample when transverse load was applied at the sample, i.e., lower bending strength. At higher sintering temperature, the diffusion rate increased and the atom diffused to adjacent grain size, the pores shrank and isolated resulting more spherical shape pores.
Figure 9. SEM image of the fractured surface of sample sintered at a rate of 5ºC/min for 30 min at 900ºC

Figure 10. SEM image of the fractured surface of sample sintered at a rate of 5ºC/min for 30 min at 1000ºC

4. Conclusion

The effects of temperature and holding time were studied during sintering of FeCrAl powder compacts formed through uniaxial die compaction process at 200ºC. The results revealed that higher relative density could be obtained by sintering the powder compacts at lower temperature, i.e., 800ºC for 30 min. However, sintering at higher temperature, i.e., 1000ºC allowed the formation of round shape isolated pores hence the bending strength of the compacts sintering at higher temperature was found to be higher. The micrographs also revealed that interconnected pores are visible at the sample sintered at lower temperature, i.e., 800ºC.

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