Heating Rate Effects on Properties of Powder Metallurgy Fe-Cr-Al_2O_3 Composites

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*Abstract***—** The aim of this study is to determine the optimum heating rate in fabricating Fe-Cr-Al_2O_3 composites by powder metallurgy methods. The Fe-based composites reinforced with ceramics are widely used due to their high strength, hardness and wear resistance. Among steps in powder metallurgy methods are mixing, compaction and sintering. Sintering is a very important step due to its ability to evolve microstructural features that govern the end properties. Sintering of green compacts made of iron powder mixture must be performed in vacuum or in a reducing atmosphere because water-atomised iron powder particles are oxidized on the surface and in this way some deoxidation reaction can occur during sintering. The heating process up to sintering temperature, plays a major role, the major proportion of densification occurs during the heating process. The composites produced were subjected to the following tests: densification, Vickers micro hardness, microstructure using SEM and X-ray diffraction analysis. From this investigation, to achieve higher densification and hardness the optimal heating rate is 10°C/minute. X-Ray Diffraction study showed that the fabrication of the composites does not lead to any compositional changes of the matrix phase and the reinforcing phase.

Keywords: powder metallurgy, heating rate, composites, densification, Vickers micro hardness.

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I. INTRODUCTION

THE development in technology is conditioned by the acquisition of new materials properties with various special functions which fulfill high different demands. Therefore a great interest in new composite materials has been observed for the last 50 years. An ability to tailor the properties of the materials to meet specific needs of an application lies in benefit of composite materials. One of the composites application areas is elements of machines, which are working in tribological connections conditions, where the following are demanded: good abrasive and wear resistance, high heat conductivity, thermal resistance and an attractive price [1-2].

The incorporation of particulate ceramics to Fe matrices significantly improve certain material properties, it offers higher hardness, higher strength at elevated temperature and wear resistance compared to monolithic Fe. Sintered Fe PM components have emerged as attractive candidates for replacing the more expensive cemented carbide and wrought alloys in many applications, due to their low cost, combination of wear resistance with toughness, high performance and ability to be processed to near-net shape. They are interesting candidate material for chemical and process industry [3-6]. When the goal is to improve the wear resistance, Al_2O_3 and Y_2O_3 particulate are used as the reinforcement on account of their hardness [7].

In this study, Fe-Cr matrix composite reinforced with Al_2O_3 was fabricated as an alternative in choosing a wear resistance material for engineering application through conventional PM method. In the economy aspect PM method is suited in manufacturing large series of small and relatively complex shapes components with smaller materials consumption [8].

Cr is added to give better corrosion resistance and to increase bonding strength of Al_2O_3. Cr is a ferrite stabilizers, it is therefore ferrite phase will be stable even at high temperature [9]. The minimum content of Cr provide a reasonable degree of corrosion resistance in Fe base alloy is 11 wt% [10].

Al_2O_3 particle is used as the reinforcement to increase friction coefficient (μ) due to their unique properties; hard and thermally stable at high temperatures, high strength with high resistance to wear and corrosion. They are important in engineering applications, such as grinding media, gas turbines, engines and solid fuel cells [11-12]. Among various ceramic particulates, good wettability of Al_2O_3 with Fe based matrix has been reported by [12-14].

The P/M process usually involves mixing of powders of the matrix alloy with the reinforcing particles, followed by compacting and solid state sintering. This typically has two heating zones, the first removes the lubricant, and the second higher temperature zone allows diffusion and bonding between powder particles. According to [15], the heating process up to sintering temperature, plays a major role, the major proportion of densification occurs during the heating process. Too low a heating rate at low temperatures dissipates driving force and results in nearly no densification, while too high a heating rate will result in distortion and warpage [16].

The goal of the present investigation was to investigate the effect of heating rate on the physical and mechanical properties of Fe-Cr-Al_2O_3 composite.

II. Experimental Procedures

In the present study, Fe powder was employed as matrix. Cr (12 wt. %) was added as alloying element to give better wear and corrosion resistance. The reinforcements used were 5 wt. % Al_2O_3 particulates. The powder mixture of the matrix alloy, the reinforcement and 2 wt% of stearic acid as a binder were blended to get a homogeneous distribution of particles in a drum shape plastic container without balls at a rotating speed of 250 rpm for thirty minutes. To make a green compact, a die of ten millimeters diameter was used. The mixed powders were uni-axially pressed at a pressure of 750 MPa. The prepared green compacts were sintered in vacuum furnace at 1100°C for two hours at (3, 5, 10, 12 and 15) °C/minutes heating rate, labeled as H1, H2, H3, H4 and H5.

Hence, the properties of the composites were evaluated. The density of each of the composites was determined using the Archimedes principle according to ASTM B311-93. Distilled water was used as the liquid for the measurements. In this technique, density is determined by measuring the difference between a specimen's weight in air and when it is suspended in distilled water at room temperature. The bulk density and percentage of apparent porosity were calculated using the following formula:

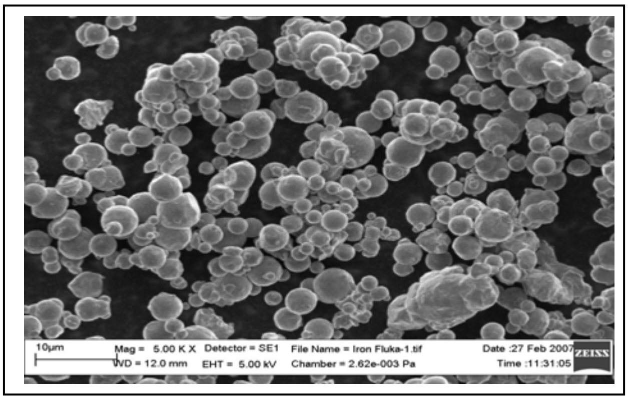
$$\text{Density}\left(\frac{g}{cm^3}\right) = \frac{W_a}{W_c - W_b} \times \text{density of water} \quad ...(1)$$

$$\text{Porosity}(\%) = \frac{W_c - W_a}{W_c - W_b} \times 100\% \quad ...(2)$$

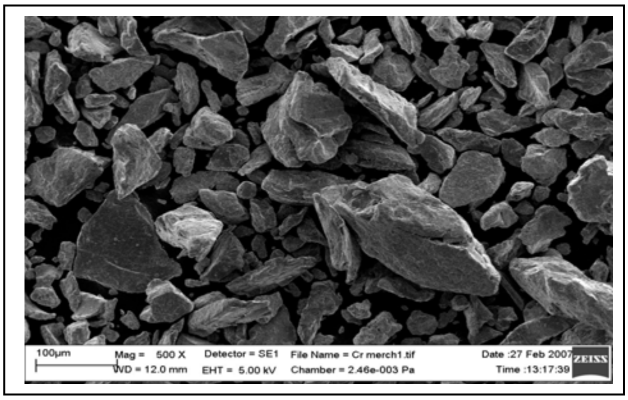
where W_a = mass of test specimen in air, g; W_b = apparent mass of test specimen, g; W_c = saturated mass of test specimen, g.

Metallographic specimens were prepared according to ASTM E3-95. The microstructures of the specimens were examined by electron microscopy and the phase analysis was carried out by X-ray Diffraction. The Vickers micro hardness data discussed in this article were obtained using a Mitutoyo Hardness Testing Machine. The value was directly determined by the size of indentation measured at ten points on the surface of the sintered composites and the average value adopted as the Vickers micro hardness of the composites.

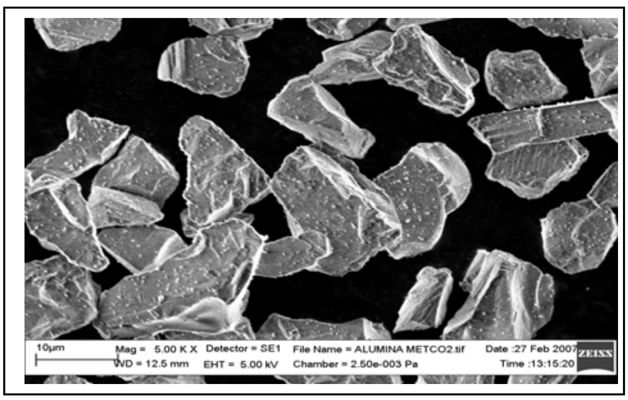
III. Result & Discussion



a) Fe powder



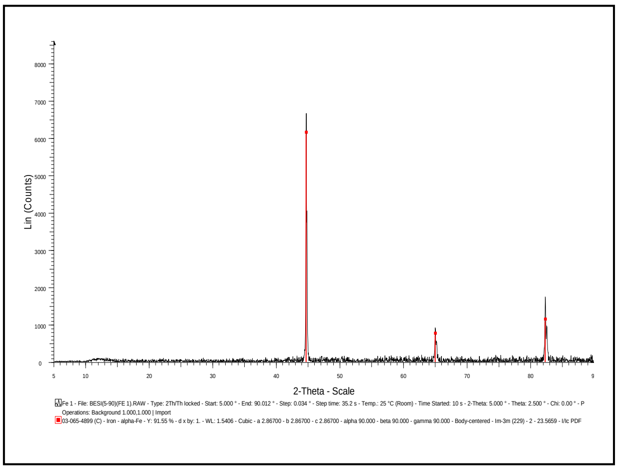
b) Cr powder



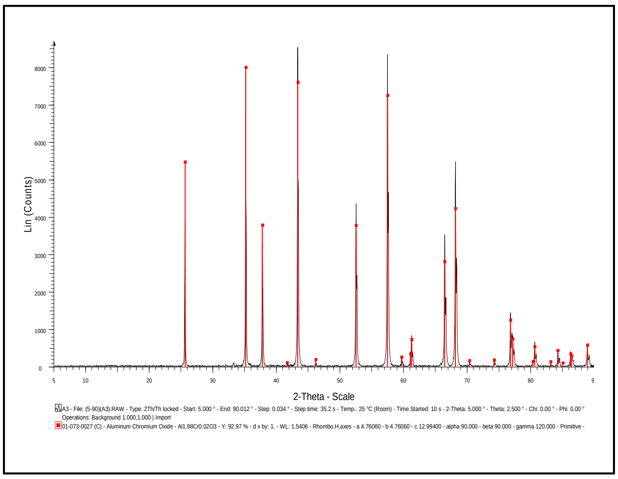
c) Al_2O_3 powder

Figure 1. SEM micrograph of raw powders .

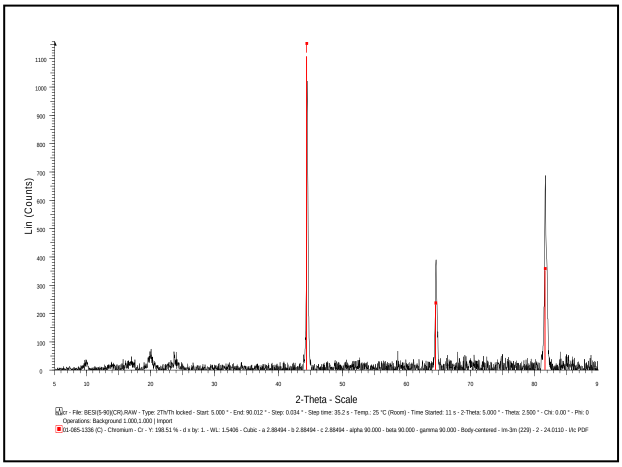
Figure 1 show the SEM of starting powder. The morphology of the as-received Fe powder is spheroidal shape with average particle size of 7.97 μm and true density is 7.2542 gcm^{-3},as shown in figure 1(a). Figure 1(b) depicted Cr powder as irregular shape and its average particle size is 25.60 μm and true density is 8.4499 gcm^{-3}. While the reinforcement powder of Al_2O_3 has an average particle size of 13.31 μm with true density of 4.6197 gcm^{-3} and it has irregular shape morphology as can be seen in figure 1(c).



a) Fe powder.



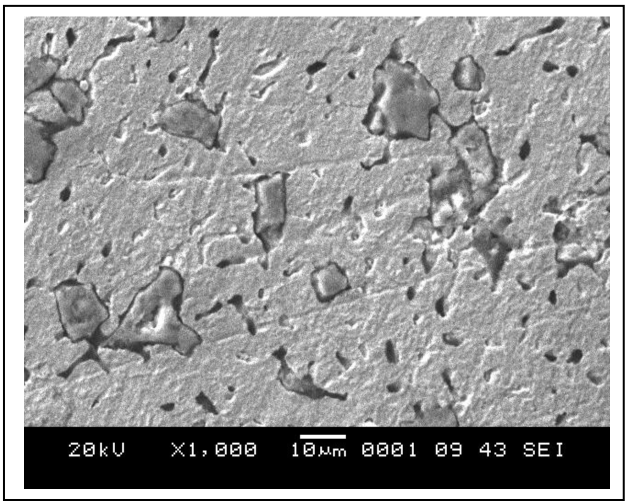
b) Cr powder.



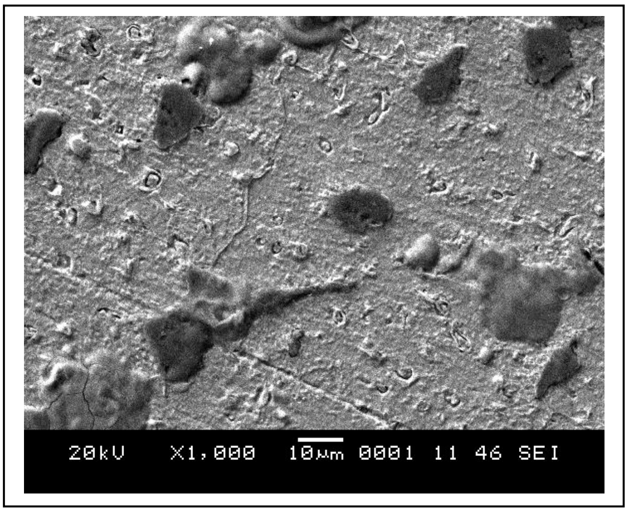
c) Al_2O_3powder.

Figure 2. XRD patterns of starting powder.

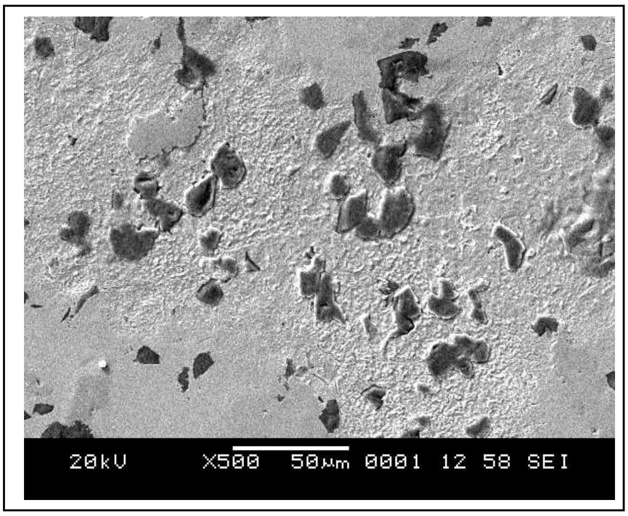
Figure 2 shows the typical XRD patterns obtained from the starting powders. Following the sequence from figure 2 (a-c), the matrix phases were identified as α-body-centered-cubic Fe with the standard number of JCPDS: 03-065-4899 and body-centered-cubic Cr (ICDD: 01-085-1336). The reinforcement particle phases were identified as corundum (ICDD: 00-010-0173).



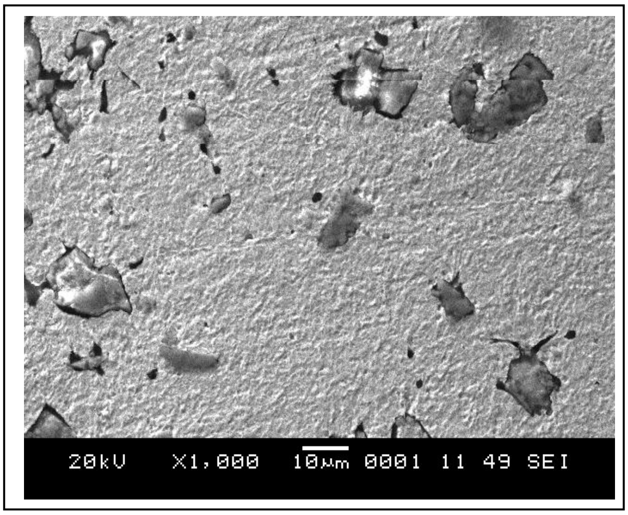
(a) Heating Rate at 3°C/min (H1).



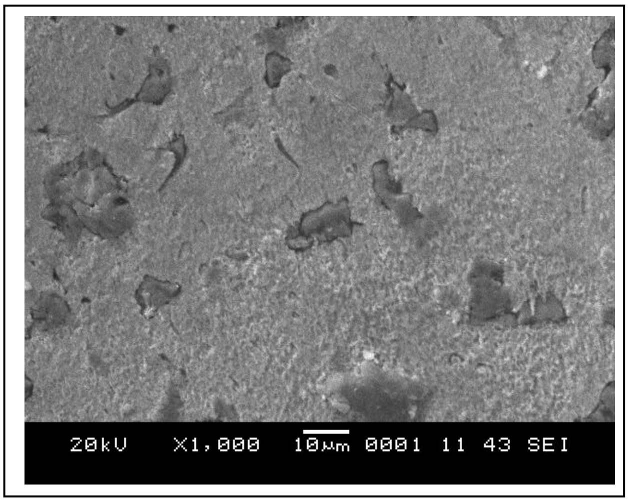
(b) Heating Rate at 5°C/min (H2).



(c) Heating Rate at 10°C/min (H3).



(d) Heating Rate at 12°C/min (H4).



(e) Heating Rate at 15°C/min (H5).

Figure 3. SEM micrograph of composite sintered at heating rate of: (a) 3°C/min, (b) 5°C/min, (c) 10°C/min, (d) 12°C/min, (e) 15°C/min.

Figure 3 show a series of composites prepared at varying heating rate. All images presented are secondary electron (SE) images captured at 1000x magnification. Observing the SEM microstructure in Figure 1(a), composites sintered at heating rate of 3°C/min have many pores and there were cavities between the reinforcement and the matrix. The microstructures were very difference than other microstructures prepared at higher heating rate.

The effect of heating rate at 5°C/min on the microstructures in figure 3(b), show that the pores were started to close and the existence of the porosity is decreasing. However the appearances of the microstructures are not satisfying because they were not smooth with cracking on the surface of the composites indicating inhomogeneous heating.

From the microstructure in figure 3(c), it can be seen that no apparent pores are formed, showing a pretty dense structure. Heating rate at 10 °C/min produced homogeneous microstructures. They did not show any sintering defects such as shrinkage cavity and cracking.

Referring to figure 3(d)-(e), increases the heating rate to 12°C/min and 15°C/min, resulted in pretty good microstructures compared to the samples sintered at lower heating rate of 3°C/min and 5°C/min. Unfortunately the results were not as good as the microstructure of composite sintered at 10 °C/min. The cavities between the Al_2O_3-Fe and the existence of porosities are quite obvious.

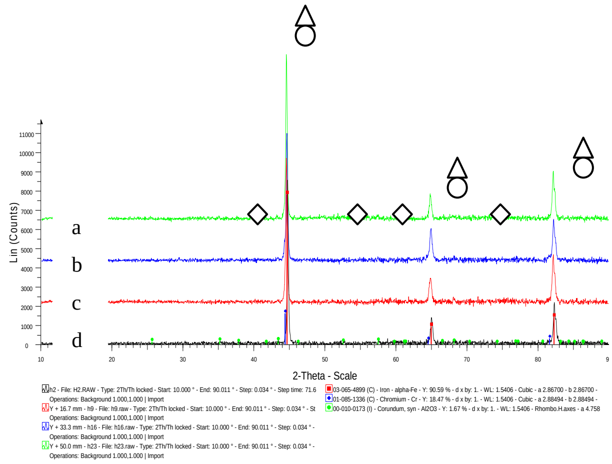


Figure 4. XRD diffractogram showing the phases of Fe( ), Cr( ) and Al_2O_3( ) in the composite sintered at heating rate of: (a) 3°C/min, (b) 5°C/min, (c) 10°C/min, (d) 12°C/min, (e) 15°C/min.

Figure 4 shows the XRD phase analysis of the composite fabricated at varying heating rate. The labels a, until d marked on each plot correspond to varying heating rate. X-ray diffraction reveals no new phases other than Fe, Cr and Al_2O_3 in the sintered composites. This may imply that there is no detectable interaction between Fe, Cr and Al_2O_3 after sintering processes. The reinforcement peaks are weak due to its relatively small content, that is 5 wt%.

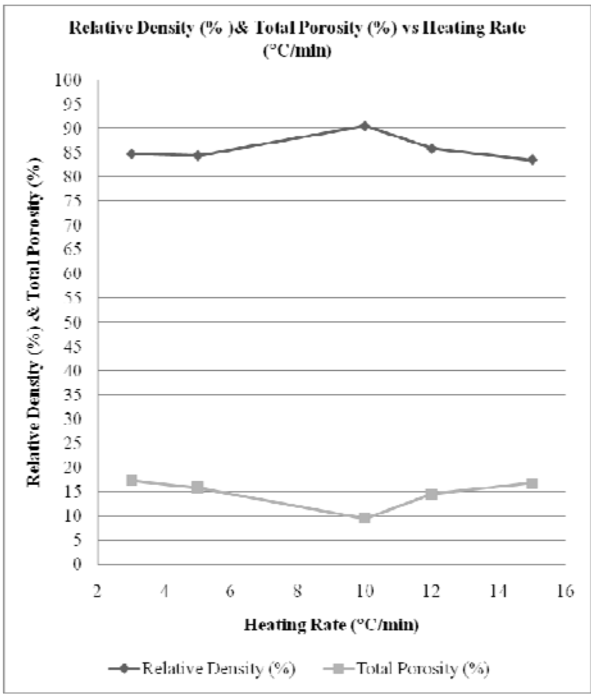


Figure 5. Percentage of Relative Density and Percentage of Total Porosity Vs Heating Rate (°C/min).

The percentage of relative density and total porosity measurements conducted on the composites samples for different heating rates are plotted in figure 5. The percentage of relative density increases and the percentage of total porosity decreases with increasing heating rate from 3°C/min until 10°C/min. Increases heating rate to 12°C/min and 15°C/min causes an increases of percentage of total porosity with decreases in relative density. The results indicate that densification is favored when the samples are sintered at 10°C/min heating rate using vacuum furnace.

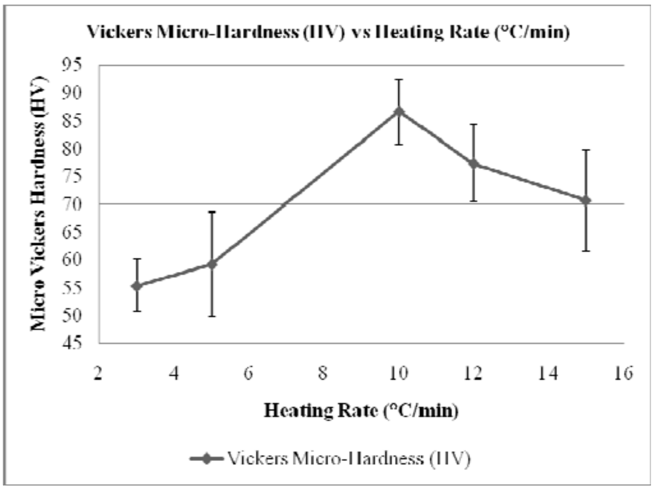


Figure 6. The Vickers micro-hardness (HV) reading of the composites sintered at different heating rate (°C/min).

The chart of Vickers micro-hardness readings conducted on the composites is shown in figure 6. By increasing the heating rate from 3 to 10 °C/min, the micro-hardness values were increasing, but further increases of heating rate until 15°C/min caused a little decreases of the reading. Micro-hardness of the composites prepared at 12 and 15 °C/min were higher than the composites prepared at lower than 10°C/min heating rate. To achieve higher densification and micro-hardness the optimal heating rate is 10°C/minute, At this heating rate thermal shock and stress gradients were avoided that might damage the powder compact.

IV. CONCLUSION

From the analysis of all the tests conducted on the composite prepared by varying heating rate, it can be concluded that the heating rate has significant effect on the microstructure, densification and micro hardness of the composites. Based on this study situation, the optimal heating rate is 10°C/minute.

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