Influence of spark plasma sintering parameters on magnetic properties of FeCo alloy

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Abstract

Equiatomic FeCo alloys with average particle size of 24 µm were sintered using spark plasma sintering (SPS) system at sintering temperatures of 1100, 800, and 850 ºC for heating rates 50, 100, 300 ºC/min by applying pressure of 50 MPa instantly at room temperature for sintering time of 5 and 15 minutes. The highest saturation induction was achieved at SPS conditions of 50 MPa, 50 ºC/min, 1100 ºC, without dwelling, of value 2.39 T. The saturation induction was improved with extending sintering time, the coercivity was higher in samples sintered at a fast heating rate in comparison to the slowest heating rate.

1. Introduction

Equiatomic FeCo alloy is one of the most important soft magnetic materials due to its high magnetic properties [1]. Spark plasma sintering (SPS) process is relatively new sintering technique with many advantages over conventional sintering processes such as high density accompanied with minimal grain growth, short time for sintering, high heating rate, high sintering temperature, binderless process, and applying pressure during sintering. This process improves different properties of various materials [2]. SPS recently used for sintering FeCo alloy composites, showing an advantage for maintaining the nanostructure features and significant improvement in mechanical properties [3, 4, 5]. Newly, the mechanical properties of FeCo alloy were significantly improved by SPS at certain sintering conditions [6]. Therefore, the current study aims to evaluate the magnetic properties of FeCo alloy sintered at different sintering conditions in order to optimise the best sintering conditions for the best combination of magnetic and mechanical properties.

2. Experimental work

2.1. Spark plasma sintering technique:

A gas atomised Fe-50Co-0.2Si alloy powder average particle size of 24 μm was supplied by Sandvik Osprey Powder Group. A twenty gram of equiatomic FeCo alloy powder was charged in a 30 mm graphite die, lined with graphite foil, which was the compacted by using a Specac manual cold press, the prepared die was then moved to the SPS furnace and exposed to a pre-programmed sintering procedure. All samples were sintered under vacuum (5 hPa), at a constant 50 MPa uniaxial sintering pressure. An optical pyrometer was inserted through a 10 mm diameter channel in the SPS tooling and into a top punch of the graphite die at a distance of 4 mm from the top surface of the sample, in order to measure the sintering temperature. Therefore, it is expected that the measured temperature is overestimated or underestimated according to the electrical property of the sintered sample with respect to die-punch assembly. The processing temperatures considered are thus insignificant. A total of three different heating rates (50, 100 and 300 $^{o}C.min^{-1}$) were investigated. Therefore, the ‘optimum’ sintering temperature was selected for each heating rate based on the point at which maximum shrinkage happened (+ 50 ^{o}C to ensure good densification). Then, the second group of samples were densified at that ‘optimum’ temperature for 15 min to assess the time taken before shrinkage accomplishes to give the ‘optimum’ sintering time. Finally, of samples were sintered for each heating rate under the ‘optimised’ sintering conditions (temperature and time). A summary of the resulting sintering conditions for all samples are seen in Table 1, the sintering conditions were given alphabetical identities for simplicity during referring hereafter.

Table 1. The identity of spark plasma conditions of sintered FeCo alloy, samples sintered at A,G,D are cited for comparison from [6].

FeCo alloy identity	Spark plasma sintering conditions		
	Sintering temperature °C	Dwelling time min	Heating rate °C/min
A	1100	without dwelling	50
B	850	15	
C	850	5	
D	1100	without dwelling	100
E	800	15	
F	800	5	
G	1100	without dwelling	300
H	800	15	
I	800	5	

2.2. Evaluation of sintered compacts:

Archimedes' immersion technique in water was used to measure the density of the sintered samples within an error of ± 0.5%. Electron discharge machining (EDM) was used to cut rectangular samples of cross section 24 × 5 mm from the 30 mm diameter sintered discs, which were ground with silicon carbide papers to eliminate the machining scratches. An automatic universal measurement system was used to evaluate the quasi DC magnetic response of the samples by adjusting the magnetic field up to 25 $kA.m^{-1}$.

3. Results and Discussion

3.1. Relative density and grain size

The relative density of FeCo alloys sintered at various sintering conditions is seen in Table 2. The density was reduced with increasing heating rates, the highest density value of 99.8 % was achieved at sintering conditions 50 $°C.min^{-1}$, 850 °C and 15 min, at sintering pressure of 50 MPa. In order to consider the influence of grain size on magnetic properties, the measured sizes are listed in Table 2. The detailed of microstructure study of FeCo alloy sintered following the above-mentioned sintering conditions are reported in our previous study on mechanical properties in [6].

Table 2 Relative density and magnetic properties of FeCo alloy at different sintering conditions.

FeCo alloy identity	Relative density (%)	Grain size (μm) [6]	Saturation induction Bsat.(T)	Coercivity Hc (A/m)
A	99.0 ± 0.4%	29.8	2.39	612
B	99.8 ± 0.4%	6.7	2.37	859
C	99 .0 ± 0.4%	6.4	2.35	896
D	99.5 ± 0.4%	23.2	2.27	435
E	99.1 ± 0.4%	6.4	2.34	875
F	97.7 ± 0.4%	6.1	2.24	961
G	99.2 ± 0.4%	22.6	2.22	473
H	98.2 ± 0.4%	6.6	2.34	850
I	96.4 ± 0.4%	5.9	2.24	956

3.2. Magnetic properties

The upper halves of hysteresis curves of sintered FeCo alloys at different sintering conditions are presented in Figure 1, a summary of the magnetic properties of the sintered FeCo alloys are seen in Table 2. Regardless of the sintering time, the saturation induction shows a linear relationship with sintering temperature for heating rate 50 ºC.min^{-1}. Inconsistent behaviour is observed for the other heating rates. The highest value of saturation induction for all sintering temperature was achieved at heating rate 50 ºC.min^{-1}, due to improved densification at this rate. The magnetic properties of FeCo alloy fabricated by powder metallurgy are mainly affected by grain size and density of the component. The former has a significant influence on coercivity, while saturation magnetisation of powder metallurgy product is more sensitive to density [7, 8]. Increase the dwelling time at a sintering temperature of 850 ºC and 800 ºC was very effective in increasing the density of the sintered FeCo alloys, therefore a significant improvement in saturation induction was achieved at these sintering conditions Table 2. Further, a change in the fraction of ordered state may happen due to sintering in lower temperature, which rationalises that even reduction in density in some samples does not because a reduction in saturation induction value since the saturation induction in fully ordered structure exceeds the disordered structure by 2-3% [9].

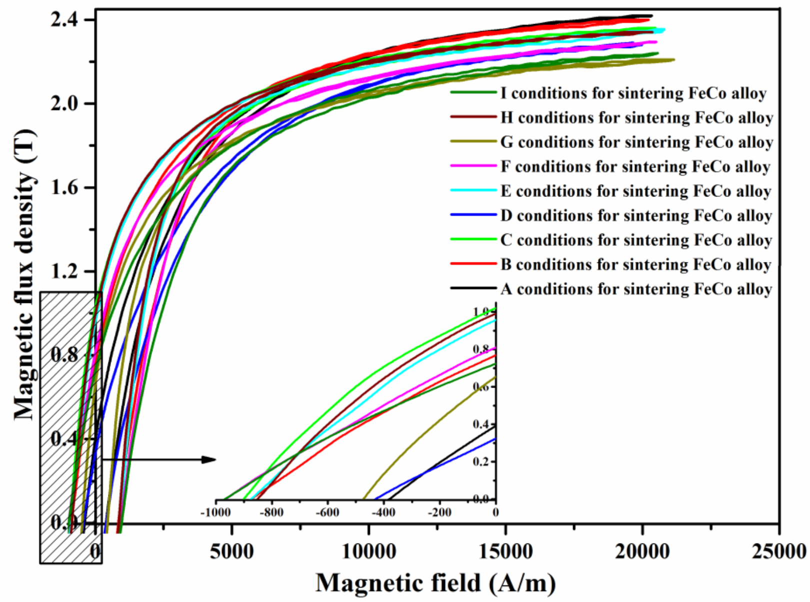

Figure 1 Upper hysteresis curves for sintered FeCo alloy at various conditions.

An increase in coercivity is observed for the samples sintered at 800 ºC of heating rates 300 ºC.min^{-1} compared with samples sintered at temperature 850 ºC for lower heating rate 50 ºC.min^{-1}. However, for all heating rates, the coercivity was lower at the highest sintering temperature of 1100 ºC [6] than other sintering temperature as shown in Figure 2. Generally, most types of defects such as grain boundaries, dislocations, precipitates effect on coercivity. Order-disorder transformation has also an influence on the final value of coercivity since the coercivity of ordered state is higher than the disordered state at room temperature [9]. The ordered structure may increase in samples sintered at 800 and 850 ºC, giving a reason for increasing coercivity in comparison to samples sintered at 1100 ºC. Furthermore, it is claimed by [1] that the coercivity is inversely proportion to grain size in the micron size order. An increase in grain size was observed at the high sintering temperature. However, the fast heating rate of 300 ºC.min^{-1} was effective in reducing grain size, therefore an increase in coercivity value was observed. Conflicting behaviour is observed when the sintering temperature is increased to 1100 ºC, due to the reduction in volume fraction of the ordered state at faster heating rates [6], and increase the grain size. Moreover, sintering at high temperature helps in releasing the coercivity dependent on residual stresses in the material which are introduced during mechanical pressing and causes more purification for structure.

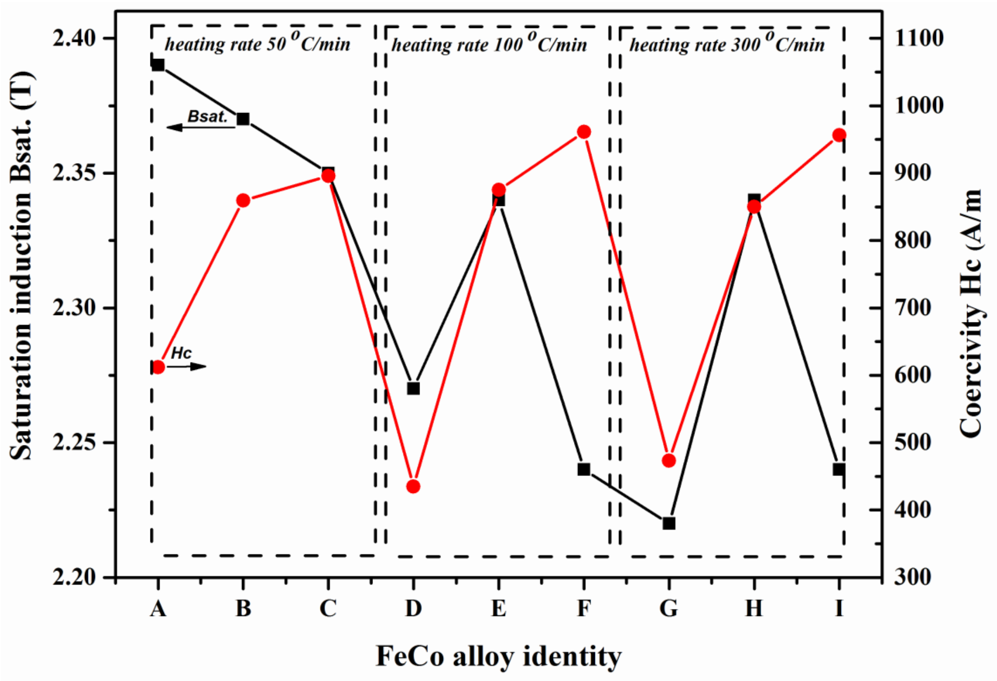

Figure 2 Summary of magnetic properties of FeCo alloy processed under various SPS conditions.

3.3. Comparison magnetic properties with alloy processed by powder metallurgy

Table 3 is a comparison of magnetic density and saturation induction of the current FeCo alloy with attempts were made by powder metallurgy PM to improve the magnetic properties of the alloy [10-12]. Spark plasma sintering process shows a significant change in magnetic properties in comparison to other sintering processes due to approximately full densification in sintered materials.

Table 3 comparisons for the density and saturation induction of the current FeCo alloy with attempts made to develop the alloy, the theoretical density for FeCo alloy is 8.17 g/cc.

Material	PM procedure	Density of material g/cc	Saturation induction (T)	Ref.
FeCo alloy	Not mentioned	8.0	1.80	[10]
Fe-50Co alloy	Cold compaction followed by sintering	95 % relative density	2.15	[11]
Fe-50Co alloy	SPS	8.14 (99.5%) relative density	2.34	[12]
Fe-50Co alloy	SPS	8.15 (99.8%) relative density	2.39	Current work

4. Summary

The SPS parameters to achieve maximum saturation induction for equiatomic FeCo alloy were found to be 1100 °C, 50 MPa, 50 ºC/min without dwelling, showing significant improvement in comparison to other PM processes to sintering FeCo alloy. The coercivity was significantly decreased at a sintering temperature of 1100 ºC due to the increase in grain size. In comparison between dwelling for heating rates 100 ºC/min and 300 ºC/min, the highest value for the relative density was only obtained by sintering at a temperature of 1100 ºC without dwelling. However, near full densification of relative density, 99.8 % was achieved by sintering at 850 ºC, 50 MPa, 15 min for the slowest heating rate of 50 ºC/min. Fast heating rates were not suitable to obtain full densification, therefore the magnetic properties were deteriorated with increasing heating rate.

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