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Temperature effect on magnetic characteristics of Fe-Cr-Co-Si alloy specimens processed by thermomagnetic treatments

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ABSTRACT

Fe-Cr-Co magnets getting research focus due to the rapid increase of magnet demand, especially for super-speed motors owing to their remanence values equivalent to that of NdFeB super magnets. In this work, the magnetic characteristics in Fe-Cr-Co-Si alloy samples were induced by processing through isothermal thermomagnetic and step aging treatments, and then, the samples were investigated for thermal effect on magnetic characteristics. The effects of an increase in temperature on the remanence, coercivity and energy product were measured using a BH-Curve tracer. The specimens were prepared from the thermomagnetically processed and aged alloy bricks. Demagnetisation curves were measured at temperatures ranging from 20 °C to 400 °C, and the results were also analysed for thermal variation coefficients. A decline in magnetic properties with increasing temperature was noted. At 400 °C, the magnetic induction is reduced by 0.03%, the coercivity by 0.018% and the energy product decreased by 0.12% per kelvin.

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Fe-Cr-Co magnets; thermomagnetic treatment; step-aging; spinodal decomposition; coercivity; magnet temperature coefficient; thermal effect; remanance

Introduction

Hard magnetic materials are always appealing for human observation for eras. The initial use of permanent magnets started with the utilisation of a magnetic compass for navigation [1]. Presently, magnets are used in a number of applications in the fields of robotics, aeronautics, communications, motors, automobiles and medical treatments.

Magnetic alloys possess unique combinations of magnetic, corrosion, mechanical, thermal and economical properties. While designing an engineering system, the characteristics of the magnet material must be considered to best suit a specific application. The desired magnetic properties include high magnetic induction, high demagnetisation resistance and high-energy products. Magnets have application for generating magnetic fields. The energy required to generate a magnetic field is stored when the magnetic material is magnetised [1]. The major role of magnets in engineering designs and devices include i) the conversion of mechanical energy into electrical energy, ii) the conversion of electrical energy into mechanical energy, iii) the transmission of electrical energy and iv) sensors. The characteristics of important permanent magnets are given in Table 1. Due to high prices and availability issues, extensive research is going on for alternatives to rare earth magnets, especially for electric vehicle motors [2,3].

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Table 1. Properties of important permanent magnets [4–6].

Magnet class	Remanence B_r, kG	Coercivity H_c, kOe	Energy product BH_{max}, MGOe	Working temp. °C	T_c, °C
Ferrites	2.00–4.60	2–5	1–5	300	450
FeCrCo	8.80–14.00	0.2–0.6	2–6	500	560
Alnico	5.50–13.70	0.5–1.8	2–10	500	800
SmCo	8.70–11.90	17–30	18–32	350	820
NdFeB	10.80–14.90	11–34	30–55	150	310

Cobalt is one of the expensive metals essentially used in the development of magnets. Due to economic reasons, the industry is now pursuing research on nonrare earth alloys, including Fe-Cr-Co magnetic alloys that can be developed with low cobalt content, as low as 10%. Fe-Cr-Co alloys possess several remarkable characteristics of very high residual magnetism, very high corrosion resistance, mechanically workable and machinable, facile production processing and low cost. In addition to these merits, magnetic characteristics equivalent to Alnico alloys can be produced, and there is potential for research on Fe-Cr-Co alloys to further enhance their magnetic characteristics. Keeping in view, its importance and the increasing need for Fe-Cr-Co magnets in high-speed drives, this research work was undertaken.

An Fe-Cr-Co magnetic alloy was initially developed by Kaneko in 1970 [7]. In these alloys, magnetic properties can be induced by the spinodal decomposition of the alpha solid solution into highly ferromagnetic alpha-1 (α_1) and weakly magnetic alpha-2 (α_2) phases [8]. The manufacturing of Fe-Cr-Co alloy is usually through melting and casting processes [9,10]. The addition of α-forming elements such as titanium, molybdenum and silicon helps to improve magnetic properties [11–13]. Research on single crystals of Fe-Cr-Co magnetic alloy indicated that magnetic properties depend on the crystallographic direction along which the magnetic field was applied during thermomagnetic treatments [14,15]. This isotropic effect of thermomagnetic treatment (TMT) gives an edge to Fe-Cr-Co alloys over Alnico alloys, as pretexturing is not required before thermomagnetic treatment. Alloying additions such as molybdenum cause to produce anisotropy in FeCrCo alloys [16]. The highest magnetic properties can be induced by applying a magnetic field in the crystallographic directions <100> during thermomagnetic treatments [17]. Studies on the processing of FeCrCo alloys by deformation aging [18] have shown that the magnetic characteristics achieved were inferior to those achieved by the TMT technique [19,20]. Zhaolong's [21] work on Fe-Cr-Co alloy demonstrated that during the spinodal decomposition of the alpha phase into α_1 and α_2, the size of the α_1-phase increased from 10 nm to 36 nm during aging treatments. The quantity of the α_1-phase increased during the early stages of the decomposition process. FeCrCo alloy with low cobalt content (~12%) and the addition of 2% silicon was thermomagnetically processed using an electromagnetic setup with a 10 kilogauss capacity. An energy product as high as 3.45 MGOe was achieved with aging treatments [22,23].

Topkaya's [24] investigation on barium ferrite magnets indicated that magnetic saturation increases with decreasing temperature. Takahashi [25] measured the effect of temperature on magnetic properties up to 500 °C, and the results indicated that iron loss decreases with increasing temperature. With increasing temperature, the magnetic flux decreases, thereby decreasing the torque of the permanent magnet motor [26]. Research studies [27–29] on NdFeB magnets have demonstrated that hysteresis loss due to microstructural imperfections may induce considerable performance loss in electrical machines. Due to their high ductility and temperature stability, FeCrCo magnets are widely applied in superspeed motors, hysteresis motors, antitheft devices and other equipment [30]. Industrial applications demand for thin and light magnetic alloys. FeCrCo magnets can be prepared into various types of filaments and thin slices via machining, deep drawing and metalworking processes [31–33]. It is possible to produce thin wires (up to 0.1 mm in diameter), thin strips (up to 0.05 mm), bars and pipes of FeCrCo alloy [34]. For FeCrCo magnets, one remaining objective is to develop a grade of FeCrCo that is equivalent in magnetic characteristics to that of Alnico magnets [30].

In this work, an FeCrCo alloy with the addition of silicon was prepared by induction melting, and the magnetic properties of the samples were induced by thermomagnetic and thermal aging processes. Samples were then investigated for temperature effect on magnetic properties. Demagnetisation curves were plotted using DC magnetometer at various temperatures from 20 °C to 400 °C. The high-temperature performance of magnets is essential for analysis, especially for their application in high-performance motors.

Experimental methodology

The manufacturing process for FeCrCo magnetic alloys starts with vacuum induction melting and casting and then the cast alloy was mechanically worked and subjected to thermomagnetic and thermal aging treatments. The FeCrCo magnetic alloy samples were processed with the following treatments:

i) Homogenisation treatment was performed under an inert argon atmosphere at 1250 °C–1300 °C for 3 h to homogenize the as-cast chemistry. After the holding period, the ingots were quenched in oil to have supersaturated alpha solid solution.
ii) Hot forging of the samples was carried out by first preheating at 1200 °C for 60 min. The temperature of forging measured by an optical pyrometer and the forging finish temperature was ensured to be not less than 1000 °C. Forging was carried out with an 80% reduction in the development of anisotropic properties. After forging the samples were air cooled,
iii) Solution treatment of the forged samples was performed under an inert argon atmosphere at 1000 °C–1100 °C for 1 h to dissolve all the alloying species to have single phase body centred cubic alpha solid solution. After the soaking period, the alloy samples were quenched in oil to have supersaturated alpha solid solutions.
iv) Thermomagnetic treatment (TMT) of the samples was performed at 5–7 kilo Gauss magnetic fields and at 670 °C–620 °C. The setup used for TMT was an electromagnetic system equipped with a resistance heating tubular furnace.
v) Step aging was performed as per Chinese patent CN101285154B in an inert argon atmosphere furnace at 620 °C/30 min, 600 °C/60 min, 580 °C/120 min, 560 °C/120 min and 540 °C/240 min.

After thermal treatments, the magnet samples were machined to final sizes and finally magnetised. The samples were characterised after thermal treatment. Chemical analysis was performed using an EDS system with a field emission scanning electron microscope. A microstructural study was conducted on the optical microscope and a scanning electron microscope. X-ray diffraction (XRD) analysis was carried out using a Panalytical Empyrean diffractometer, where the samples were scanned from 20° to 90° 2-θ range. Thermal analysis was performed using a NETZSCH STA 449 DTA analyzer. Magnetic properties were measured using Dexing BH-Curve tracer FE2100H.

Results and discussion

Chemical analysis of the developed FeCrCo alloy is given in Table 2. The cobalt content to around 13 wt.%, silicon is 2 wt.%, 0.6 wt.% titanium and 0.85 wt.% vanadium were added to the alloy. The addition of aluminium (~0.3 wt.%) was made to stabilize the alpha-phase and avoid undesired phases.

Metallography

Metallographic analysis revealed that grains of size in the range of 50–150 microns were formed, as shown in Figure 1A and B. A scanning electron micrograph, as shown in Figure 1C, demonstrates the formation of an α_1 fine spike-like structure. The length of the α_1 particle is around 167 nm, and the width is around 30 nm. The structure containing α_1 particles in the α_2 matrix is formed during the spinodal decomposition of the BCC alpha phase. The alpha-1 phase is rich in FeCo and is highly magnetic, while the alpha-2 phase is rich in chromium and weakly magnetic. In FeCrCo-type alloys, a two-phase structure is formed by thermomagnetic and step-aging treatments, whereby strong magnetic properties are induced in the alloy.

Table 2. Chemical analysis of FeCrCoSi alloy sample (weight percent).

Fe	Cr	Co	Si	V	Ti	Al
58.25	24.94	13.06	2.00	0.80	0.63	0.32

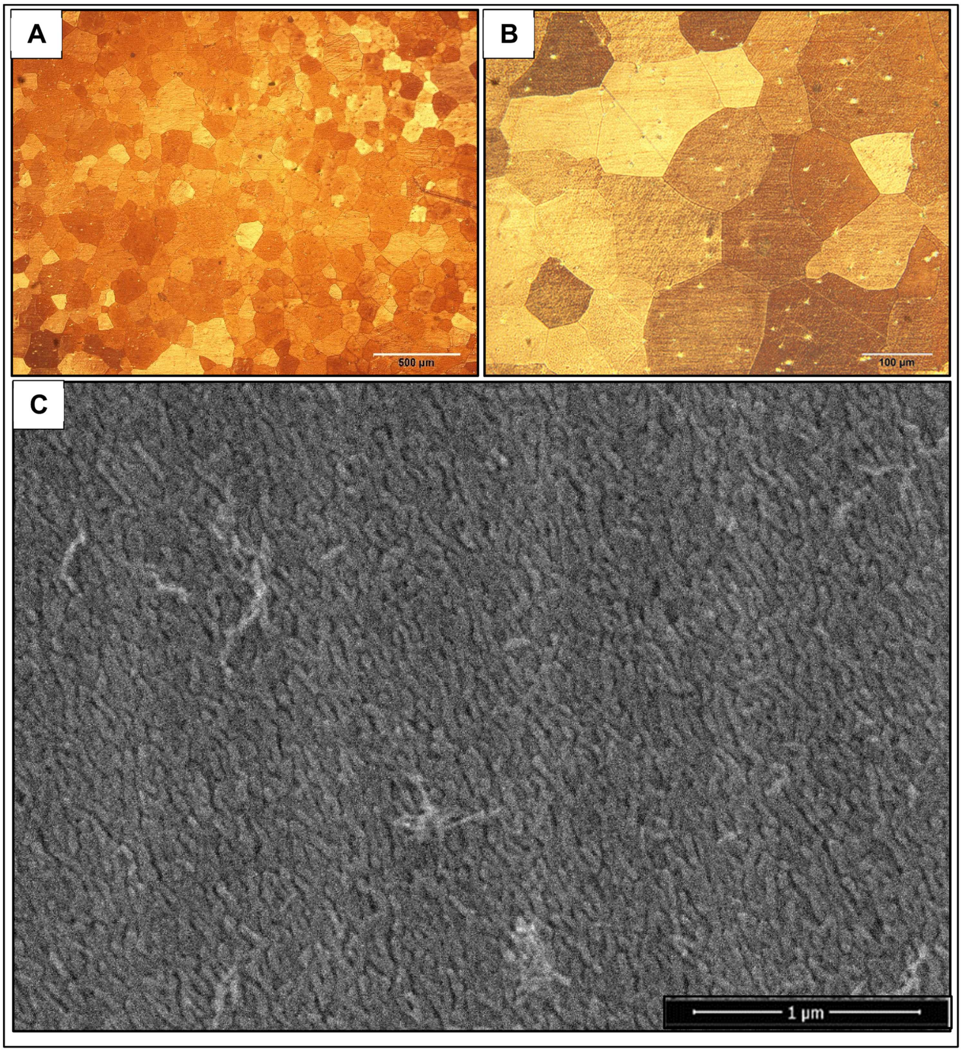

Figure 1. (A and B): Optical micrographs showing the alpha (α) grains of 50–100 microns, (C): scanning electron microscope micrograph revealing α_1 phase having length of 167 nm and width around 30 nm.

Kaneko [35] revealed that the Fe-Cr-Co alloy system when doped with tungsten, molybdenum and silicon shows enhanced magnetic characteristics that are comparable to those of well-known Alnico magnets.

X-ray diffraction (XRD) analysis

XRD analysis result graphs are shown in Figure 2. The graphs demonstrate that the main phase in the FeCrCo sample is a body-centred cubic (BCC)-type structure. The major X-ray peaks are matched with the PDF card 96-152-4270. The (110) peak of the BCC ferrite phase is noticeable. The highest peaks for alpha-1 and alpha-2 (Figure 2) indicate that spinodal decomposition occurred properly during the thermo-magnetic and aging treatments.

The magnetic properties of silicon with added Fe-Cr-Co magnets were also investigated by Szymura [36], who found that Si-addition prevents α to γ phase transformation. Silicon reacts with oxygen to develop metal oxides, which reduce the critical cooling rate that ultimately prevents α to γ transformation. The addition of silicon also increases the magnetic moment by filling 3d-subshell voids by valence electrons. This action of silicon helps to enhance magnetic remanence and also energy product of the alloy system. Phase separation of α_1 and α_2 phases within the miscibility gap of the alloy system initiates at low supersaturations by the nucleation process. However, at high supersaturation rates, unmixing occurs by spinodal decomposition process [37,38]. During the process of spinodal decomposition, the

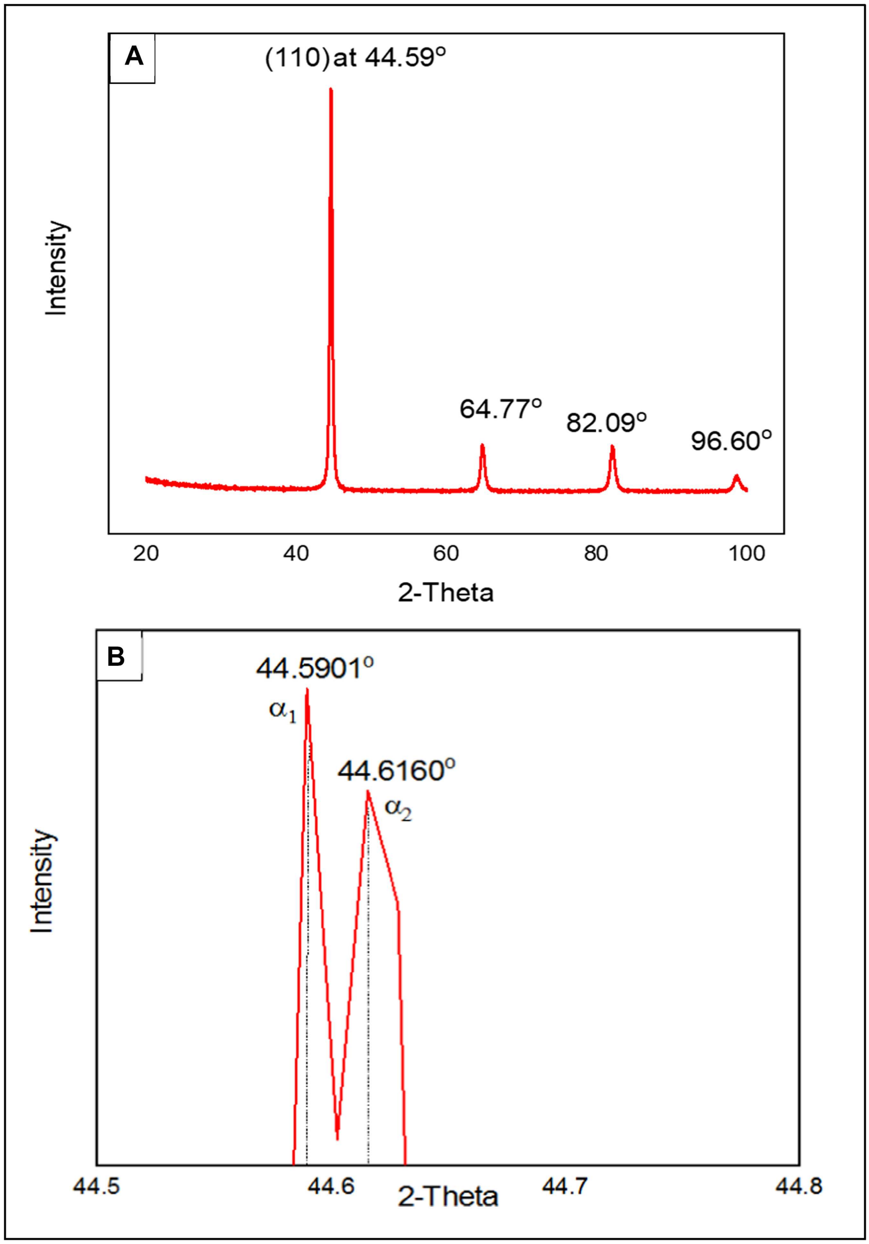

Figure 2. X-Ray diffraction pattern of the Fe-Cr-Co sample, A) showing the formation of BCC ferrite phase, B) the formation of α_1 and α_2 phases.

composition of the emerging phases changes progressively with aging treatment time until equilibrium in the phases is attained. Coarsening of the alpha-1 phase structure occurs with aging treatment time. The magnetic field intensity and temperature during thermomagnetic treatment and the step-aging treatment regime are the significant factors to affect the magnetic properties.

Differential thermal analysis (DTA)

The transition and transformation temperatures are essential for designing the effective thermal treatments. Any change in the chemical composition of FeCrCo alloy causes changes in the transition temperatures, spinodal decomposition temperatures and Curie temperatures. Differential thermal analysis (DTA) was carried out to determine the effective temperatures where transformations in typical FeCrCo alloys take place. Alloy addition affects spinodal transition temperatures; therefore, the phases formed, especially during the thermomagnetic treatments, are influenced. Thermal analysis is therefore essential for the preparation of heat treatment cycles.

Differential thermal analysis (DTA) curves for the Fe-Cr-Co sample are shown in Figure 3. In the graph in Figure 3, the first peak appeared at 587 °C. According to the FeCrCo alloy phase diagram [39], this peak corresponds to the $\alpha \rightarrow \gamma$ phase transformation, which means that the BCC structure is transformed to the FCC structure. The DTA curves demonstrate that the exothermic behavior continues at 780 °C. The exothermic behavior is due to the continuous increase in the quantity of the γ-phase with increasing temperature. The cooling curve also confirms the transformation of the γ-phase to the α-phase at 612 °C. The measurement of these transformation temperatures is helpful in designing thermomagnetic treatment regimes.

Magnetic properties

The magnetic characteristics of the FeCrCoSi samples were tested at various temperatures in the range of 20 °C–400 °C. Heating was performed by using a tube furnace equipped with an inert argon atmosphere where the samples were placed in a ceramic housing to ensure that the temperature was constant during the measurements.

The temperature of the samples was monitored by using an infrared temperature reader. The heated sample was placed within the sensing coil between the two poles of the Dexing DC magnetometer. The sensing coils of the Gauss meter and Flux meter give values of magnetic induction as a magnetic field was applied to the sample. Hysteresis curves were plotted, as shown in Figure 4A, and the values of magnetic remanence, coercivity and maximum energy product were measured for each test. Demagnetisation curve plots obtained at test temperatures ranging from 20 °C to 400 °C are shown in Figure 4B. The results for the change in magnetic characteristics with increasing temperature are given in Table 3. It can be seen from the results that magnetic induction (B_r) decreased from 14.13 kG at 20 °C to 12.25 kG at 400 °C. The coercive strength (H_c) of the samples decreased from 570 Oe at 20 °C to 530 Oe at 400 °C. The maximum energy product (BH_{max}) decreased from 5.27 MGOe at 20 °C to 2.74 MGOe at 400 °C. It can be observed that the decrease in coercivity of Fe-Cr-Co magnetic samples is very little, only 7% compared to that of the energy product. The decrease in remanence is also very small and not significant. The results demonstrate

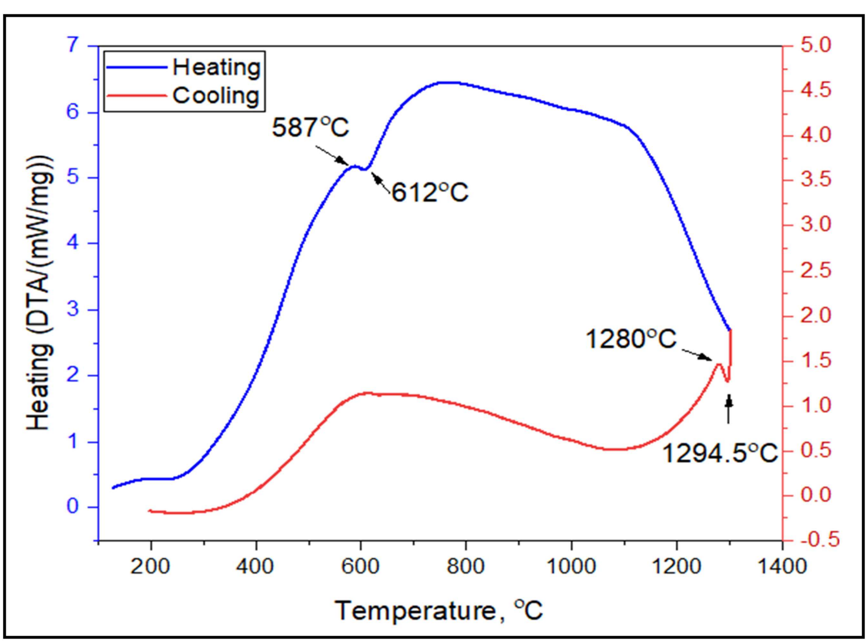

Figure 3. Differential thermal analysis for the Fe-Cr-Co-Si alloy sample showing thermodynamic activities during and heating and cooling cycles.

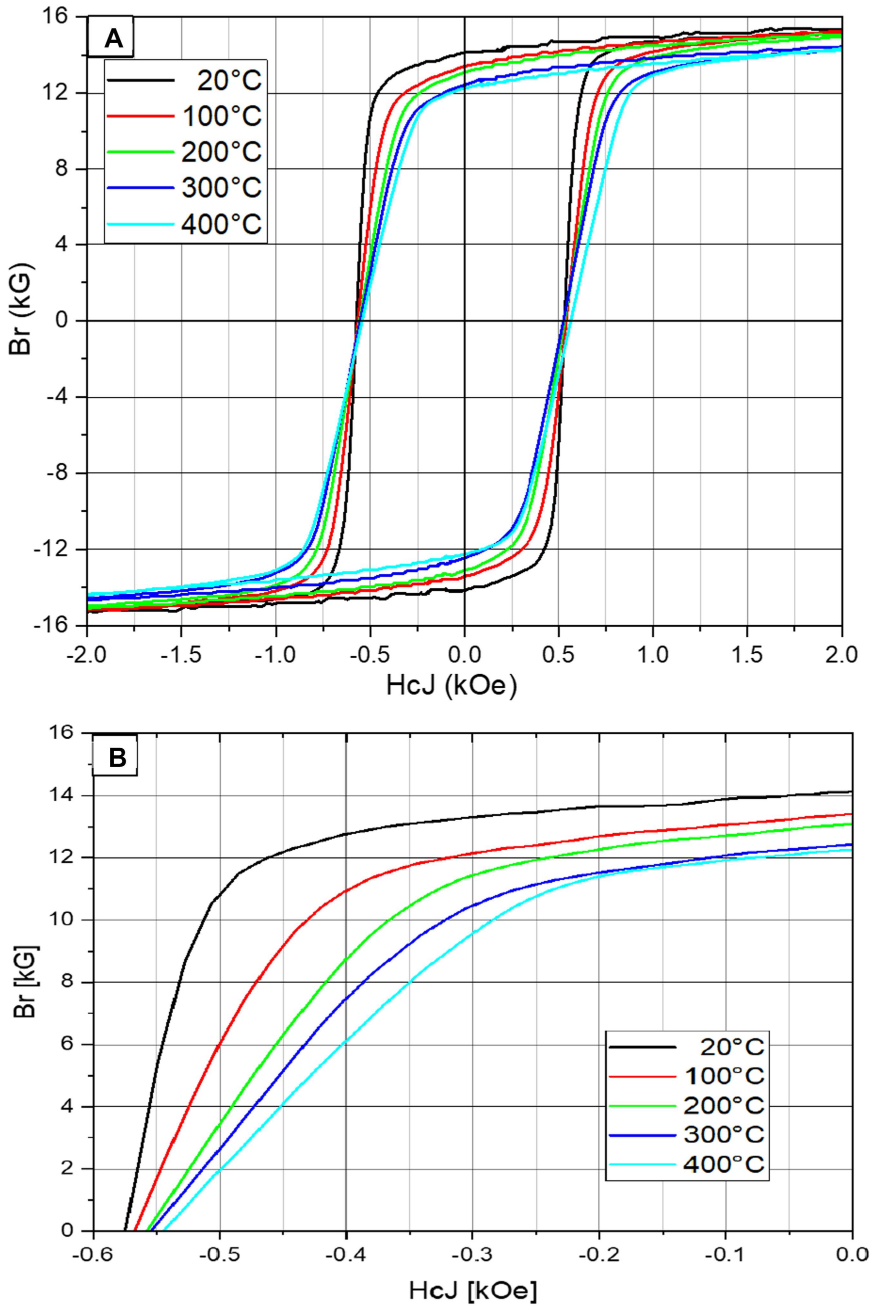

Figure 4. A) BH curves showing the coercivity and remanence for the Fe-Cr-Co-Si samples tested at 20 °C to 400 °C range, B) comparison of demagnetisation curves of the samples.

Table 3. Results of magnetic tests of Fe-Cr-Co-Si samples at given temperatures.

Test	Temperature	Br, [kG]	Hcj, [Oe]	BH_{max}, [MGOe]
1	20 °C	14.13	570	5.27
2	100 °C	13.41	560	4.14
3	200 °C	13.09	550	3.49
4	300 °C	12.43	540	3.08
5	400 °C	12.25	530	2.74
Overall decline in properties		13.31%	7.02%	48.01%

that the FeCrCo magnet can perform well without being demagnetised at elevated temperatures up to 400 °C.

The data show that an increase in temperature has a significant effect on the magnetic energy product of the Fe-Cr-Co-Si samples. Notably, the energy spectrum at the magnetic domain level is bound with the

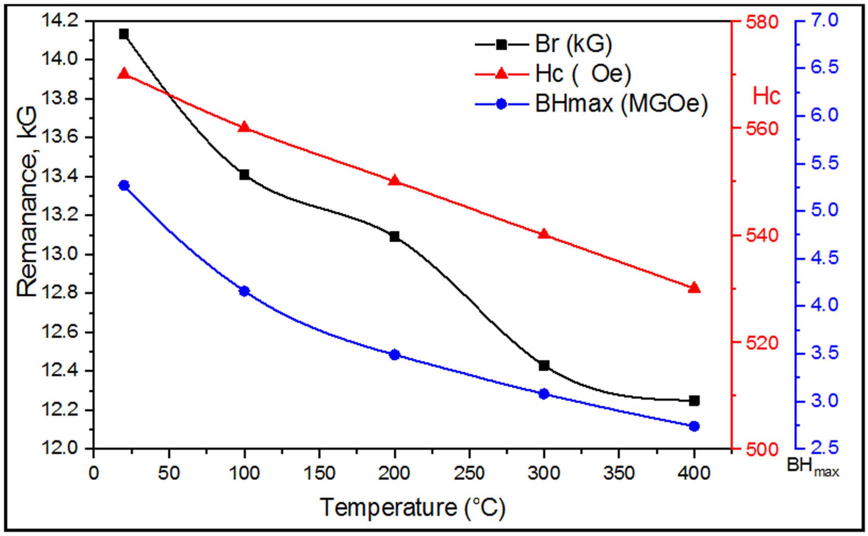

Figure 5. Magnetic properties change with temperature for FeCrCoSi magnet.

Table 4. Calculation for the TVCs for Br, Hc and BH_{max} of FeCrCoSi magnet.

Characteristic	TVCs formula	Thermal coefficient %/K (mean)
Remanence, B_r	$\alpha_{Br} = \frac{1}{Br}\frac{\Delta Br}{\Delta T}\,100\%$	−0.0350
Coercive magnetic field, H_c	$\alpha_{Hc} = \frac{1}{Hc}\frac{\Delta Hc}{\Delta T}\,100\%$	−0.0185
Maximum energy product, BH_{max}	$\alpha_{(BH)max} = \frac{1}{(BH)max}\frac{\Delta (BH)max}{\Delta T}\,100\%$	−0.1263

equilibrium of atomic vibrations. The atomic vibrations increase with increasing ambient temperature and interrupt the domain alignment, causing a decline in magnetic properties. The magnetic characteristics are affected by an increase in temperature causes to misalign the magnetic domains. The performance of the Fe-Cr-Co magnets is also affected at elevated temperatures; therefore, any decline in properties can be measured and accommodated while designing engineering applications. Data for the magnetic properties (B_r, H_c and BH_{max}) obtained from various temperature tests were plotted (Figure 5), demonstrates variation in the magnetic properties with increasing temperature. The magnetic remanence and maximum energy product both start decreasing with increasing temperature; however, the coercivity values are almost stable up to 400 °C. The results indicate that the Fe-Cr-Co-Si samples do not fully demagnetize up to 400 °C. A slight decrease in magnetic induction indicates that if the magnet is utilised in hysteresis motors, the performance of the motor would not decline much up to 400 °C. As the energy product BH_{max} is decreased by 48%, the pull force of the magnet should also be reduced proportionally; therefore, in applications where the pull force is the prime property, the BH_{max} values of the FeCrCo magnet at working temperatures must be considered while designing its application.

The effect of temperature is usually quantised in terms of the 'temperature variation coefficient (TVC)' for a magnet. The temperature coefficients are utilised to characterise a magnet for its temperature dependence [40]. The temperature variation coefficients describe the loss in magnetic values with increasing temperature. In general, at higher temperatures, magnets lose their magnetic characteristics. By measuring the temperature variation coefficients, the performance of magnets at higher working temperatures can be established. The temperature variation coefficients for the remanence (Br), coercivity (Hc) and maximum energy product (BH_{max}) for the FeCrCoSi samples were computed from the test data, as shown in Table 4. Temperature variation coefficients for Fe-Cr-Co magnet samples as calculated are with negative signs, which indicate that the magnetic characteristics decrease with increasing temperature. The data in Table 4 demonstrate that all the magnetic properties weaken and magnets lose their characteristics with increasing test temperature up to 400 °C. The weakening in B_r, H_c and BH_{max} is estimated to be 0.035%, 0.018% and 0.126% per kelvin, respectively.

Conclusions

Fe-Cr-Co-Si magnetic alloy samples were characterised, and the effects of temperature on magnetic characteristics were investigated. Phase analysis confirmed the formation of alpha-1 and alpha-2 phases as a result of spinodal decomposition. Microscopy demonstrated the formation of a spike-like alpha-1 (α_1) magnetic phase with a size of around 30 nm. Thermal analysis demonstrated that the phases formed are stable below 587 °C. The magnetic characteristics (remanence, coercivity and energy product) were measured at temperatures of 20 °C–400 °C. A decrease in magnetic characteristics was noted with increasing test temperature. At 400 °C, the decrease in B_r, H_{cj} and BH_{max} was found to be 13%, 7% and 48%, respectively. Thermal vibrations are associated with this phenomenon. An increase in thermal vibrations interrupts the domain alignment and causes a decrease in magnetic performance. The magnet temperature coefficients indicate the magnet performance with increasing temperature. The results indicated that for Fe-Cr-Co-Si magnets, the magnetic induction was reduced by 0.03%, the coercivity decreased by 0.018% and the energy product BH_{max} decreased by 0.126% per kelvin. This investigation demonstrated that the FeCrCoSi magnets can perform well at elevated temperatures up to 400 °C with very little variation in magnetic performance.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

The data supporting the findings in this work are available from the corresponding author on request.

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