

Microstructure Dependence of Magnetic Properties for $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ Multi-Principal-Element Alloy

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Abstract: This study focuses on the microstructures and soft-magnetic properties of the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ multi-principal-element alloy (MPEA) in different states. The MPEA was prepared using arc melting and suction-casting, followed by various heat treatments. The crystal structures were analyzed using X-ray diffraction (XRD), while the microstructures were characterized by means of transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The results reveal that the MPEA consists mainly of coherent body-centered cubic (BCC) and B2 phases, with a moderate lattice misfit ($\varepsilon = 0.14\sim 0.21\%$) between them. The homogenized alloy shows the presence of coarse equiaxed grains and micro-scale cells, and it has good soft-magnetic properties with $M_s = 127$ emu/g and $H_C = 143.3$ A/m (1.8 Oe). The thermal stability of the alloy is found to be optimal after aging at 873 K, as there are no significant changes in microstructures and soft-magnetic properties. However, when the aging temperature increases to 973 K, the BCC nanoprecipitates are coarsened, leading to a decrease in the soft-magnetic properties.

Keywords: multi-principal-element alloys; $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ alloy; soft-magnetic properties; coherent precipitation; microstructural stability



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1. Introduction

Soft-magnetic materials are widely applied in various fields because of their low coercivity (H_c), high magnetic conductivity, and high saturation magnetization (M_s or B_s), such as power transformers, electronic circuits, and information storage [1,2]. High-temperature soft-magnetic materials (operating temperature range from 673 K to 1073 K) are especially critical for developing advanced weapon systems and aerospace and aviation industries. They can be utilized as stable power switches, high-frequency inductors, transformer cores, engine rotors, magnetic shafts, or magnetic material devices [3,4]. Traditional soft-magnetic materials, such as silicon steel sheets, Fe–Ni/Fe–Co alloys, amorphous and nanocrystalline soft-magnetic alloys, and soft-magnetic ferrites, suffer from brittleness, complex manufacturing processes, low electrical resistivities resulting in very high eddy current losses, and poor thermal stability resulting in low operating temperatures [5,6]. With the increasing demand for high-performance soft-magnetic materials in harsh environments such as high-speed rotation, torsion, and vibration, it is vital to develop new soft-magnetic materials with high electric resistance, saturation magnetization, low loss, low coercivity, high thermal stability, and prominent strength and ductility [7–9]. However, traditional alloy design methods cannot meet these requirements due to the difficulty in achieving both high strength and excellent soft-magnetic properties simultaneously [10].

In recent years, multi-principal-element alloys (MPEAs), also named high-entropy alloys (HEAs), have obtained significant attraction due to their unique properties and potential applications [11,12]. Unlike traditional alloys, which typically consist of one or two dominant alloying elements, MPEAs can achieve a synergistic design of mechanical

and functional properties by utilizing what seems like an infinite composition space, such as high hardness and wear resistance, good corrosion resistance, excellent radiation resistance, and damping properties [13–15]. MPEAs enriched in 3D transition metal elements, such as Co, Fe, Ni, Cu, or Mn, have demonstrated considerable potential for soft-magnetic applications [11,14–16]. In the realm of such alloys, a series of FeCoNi-based MPEAs has been developed in recent years and is notably significant, exhibiting superior mechanical and ferromagnetic properties [16–19]. Among them, FeCoNi(AlSi) x alloys achieved a saturation magnetization of $M_S = 125$ emu/g and a coercivity of $H_C = 1400$ A/m [20,21], whereas the FeCoNi(CrAl) x composite exhibited saturation magnetization values ranging from 13 to 64 emu/g and a coercivity of 1416 A/m [16]. In addition, a large amount of L_{12} nanoparticles was precipitated into the FCC matrix in the $\text{Fe}_{32.6}\text{Co}_{27.7}\text{Ni}_{27.7}\text{Ta}_{5.0}\text{Al}_{7.0}$ alloy, which is regulated to a particle size of 91 nm, providing relatively low coercivity since the size of these L_{12} nanoparticles is less than that of the domain wall, and yet sufficiently large to reduce coherency distortions on ultrafine nanoparticles [22,23].

Although the above research has illuminated the remarkable promise of MPEAs in soft-magnetic devices, there are still important scientific questions that remain to be answered. In our previous work, we designed a soft-magnetic $\text{Al}_{1.5}\text{Co}_4\text{Fe}_2\text{Cr}$ alloy through the cluster-formula approach, adopting the formula of Al_3M_{14} , where M can be set and represents different combinations of Co, Cr, and Fe. This alloy exhibited the marked properties of $M_S = 135.3$ emu/g, $H_C = 127.3$ A/m, $T_C = 1061$ K, and $\rho = 244$ $\mu\Omega\cdot\text{cm}$, respectively [24–26]. Its prominent soft-magnetic properties could be attributed to its distinct coherent microstructure, characterized by spherical, ultrafine, and ferromagnetic body-centered-cubic (BCC) particles with a size of 4–8 nm, homogeneously dispersed within the B2 matrix. Nevertheless, once the BCC/B2 coherent microstructure was in reverse, i.e., large cuboidal B2 nanoprecipitates (~100 nm) were precipitated into the BCC matrix in the $\text{Al}_{0.7}\text{NiCoFeCr}_2$ alloy developed via the cluster formula of Al_2M_{14} , the soft-magnetic property was severely deteriorated, indicated by a reduced saturation magnetization of $M_S = 28.9$ emu/g and an increased coercivity of $H_C = 947$ A/m. Furthermore, the presence of a weave-like BCC/B2 coherent microstructure in $\text{Al}_{0.57}\text{NiCoFeCr}$ alloy leads to an almost loss of the soft-magnetic feature, as evidenced by the much higher $H_C = 4035$ A/m and very low $M_S = 11.7$ emu/g [24,26]. These findings showed that the magnetic properties of MPEAs are critically sensitive to the microstructure. Moreover, the influence of the microstructural evolution of BCC/B2 with heat treatment on the soft-magnetic properties should be further investigated in depth. Therefore, in this study, we adopt the cluster formula of Al_3M_{14} to design a new MPEA in the Al–Co–Cr–Fe system. The cluster formula approach has been used to design alloy compositions in complex composition systems, enabling rational matching of the alloying elements and thus determining the amount of each element [24–26]. More importantly, the BCC/B2 coherent microstructure with spherical or cuboidal nanoparticles could be well governed by the cluster formula in a complex composition alloy containing Al and transition metal elements to obtain outstanding mechanical and functional properties simultaneously [17,25–27]. Under the above considerations, the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA was obtained, and the microstructure and magnetic properties of this alloy in different treatments were examined and tested to investigate the influences of microstructural evolution (including phase constitutions and particle morphologies) on the soft-magnetic properties.

2. Materials and Methods

The $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA was prepared through arc melting and suction-casting into a copper mold with a dimension of 2 mm $\times$ 9 mm $\times$ 60 mm under an argon atmosphere. The purity of raw metals exceeds 99.99 in weight percent (wt. %). To achieve chemical uniformity, approximately 10 g of the alloy ingots was melted and remelted at least five times prior to being cast by suction. Following this process, the cast alloy sheets underwent a homogenization treatment at 1573 K for 2 h and were then subjected to aging for 24 h at varying temperatures (773, 873, and 973 K). In a particular set of experiments aimed at investigating the microstructure's thermal stability, selected alloys were aged at tempera-

tures between 873 K and 973 K for an extended period of 480 h. All specimens were rapidly cooled in water subsequent to the thermal treatments.

Investigations into the crystal structures of the homogenized and aged alloys were conducted employing a Bruker D8 X-ray diffractometer (XRD) (Bruker Ltd., Fremont, CA, USA) with Cu- K_{α} radiation ($\lambda = 0.15406$ nm) at a scanning speed of $2^{\circ}/\text{min}$, during which the lattice constants of phases were calculated via an external standard method [28]. The detailed characterization of microstructures was carried out by means of a Zeiss Supra 55 scanning electron microscope (SEM) (Zeiss Ltd., Jena, Germany) with an electron acceleration voltage of 15 kV and JEOL-JEM-2100F field-emission transmission electron microscope (TEM) (JEOL Ltd., Tokyo, Japan) with an electron acceleration voltage of 120 kV. To further analyze the morphology of ultrafine nanoprecipitates and elemental distribution, an aberration-corrected JEM-ARM300F TEM (JEOL Ltd., Tokyo, Japan) equipped with an energy dispersive spectroscopy (EDS) detector with an electron acceleration voltage of 200 kV was employed. Sample preparation for SEM analysis involved a series of mechanical processes: initial grinding followed by polishing, and a final etching in a solution consisting of 5 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 25 mL HCl, and 25 mL $\text{C}_2\text{H}_5\text{OH}$. TEM specimens were prepared using an FEI Helios NanoLab 600 Dual-Beam focused ion beam (DB-FIB) instrument (FEI Ltd., Hillsboro, OR, USA), the detailed procedure being described elsewhere [29]. The statistical analysis of the particle size of nanoparticles was performed with at least six SEM or TEM morphology images using Image-Pro Plus 6.0 software. Magnetic properties, specifically saturation magnetization and coercivity of the alloys in different states at room temperature, were measured using a vibrating sample magnetometer (VSM, Lake Shore 7410) (Lake Shore Ltd., Columbus, OH, USA) with a maximum applied field of 15,000 Oe. Compared to other techniques like Alternative Current Magnetic Susceptibility [30] or Magnetic Force Microscopy [31], VSM can not only measure non-contact magnetic field strength, avoiding the problem of electromagnetic interference in traditional measurement methods but also requires less of a sample and is not limited by the shape and size of the sample.

3. Results and Discussion

3.1. Microstructure of $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA in Different States

Figure 1 shows the XRD patterns of the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA in homogenized and aged conditions. The XRD analysis reveals that all the different heat-treated alloys are mainly constituted of BCC and B2 phases. The B2 phase is characterized by a weak peak (100) diffraction plane appearing at $2\theta \sim 31^{\circ}$ after homogenizing, which becomes much more markedly pronounced when the alloy undergoes aging at 773, 873, and 973 K for 24 h. The lattice misfits between BCC and B2 phases in both solid-solutioned and aged alloys are calculated by the equation $\varepsilon = 2 \times (a_{\text{B2}} - a_{\text{BCC}}) / (a_{\text{B2}} + a_{\text{BCC}})$, as presented in Table 1. The lattice misfits of these RHEAs in different states are in the range from 0.14% to 0.21%. It is worth noting that these ε values are relatively smaller, compared with those ($\varepsilon = 0.2 \sim 0.6\%$) in previous studies on Al–Ni–Co–Fe–Cr MPEAs exhibiting BCC/B2 coherent microstructures [25,26].

Microstructures of these homogenized MPEAs were characterized by Optical Micrograph (OM) and SEM. Figure 2a shows the OM morphology of $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ alloys, which are composed of coarse equiaxed grains with an average size ranging from 200 to 400 μm . Further examination using SEM in a homogenized state, as shown in Figure 2b, demonstrates that each grain comprises micro-scale cells measuring between 120 and 400 nm. The emergence of micro-scale cells in this alloy can be attributed to the preferential segregation of Fe and Co elements along the cell boundaries [24].

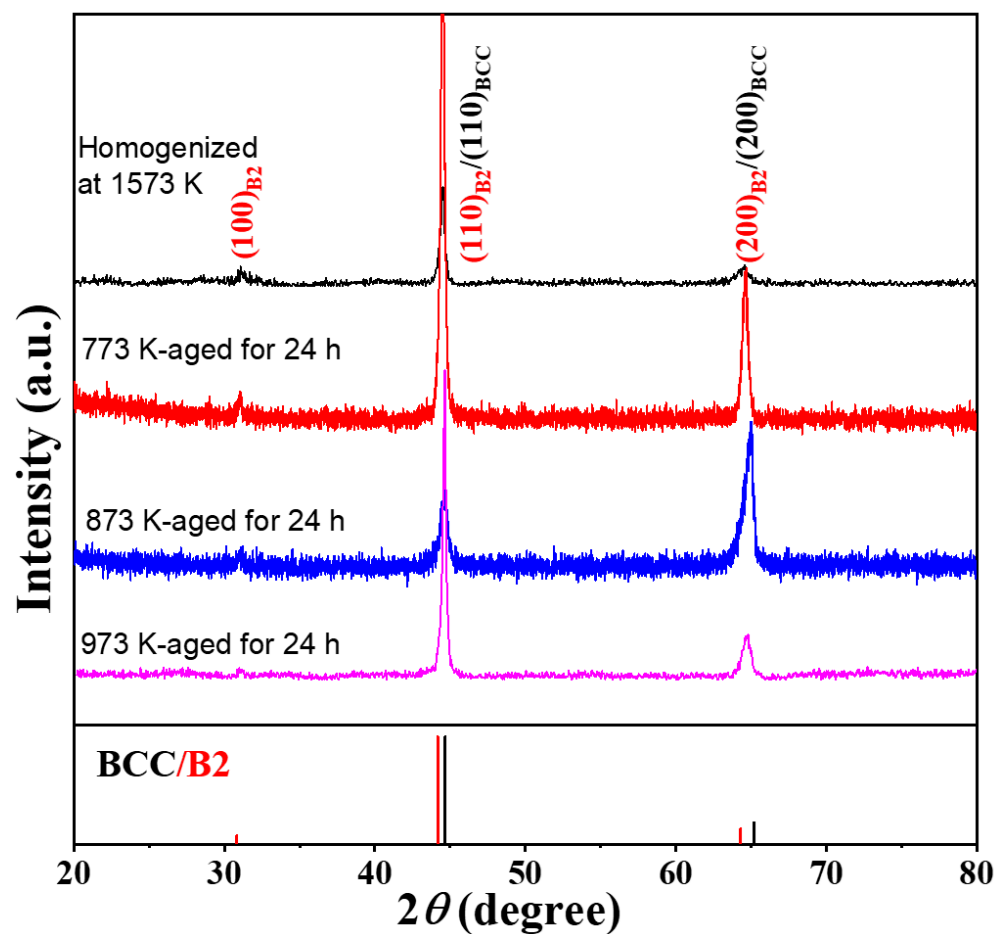


Figure 1. The XRD patterns of the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA in homogenized and aged states.

Table 1. Data summary for the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA, including the chemical compositions in atomic percent (at. %) and magnetic properties (characterized with the saturation magnetization M_S and coercivity H_C) in different heat-treated states.

Alloy	Composition (at. %)	Heat Treatment	M_S (emu/g)	H_c	
				(Oe)	(A/m)
$\text{Al}_{1.5}\text{Co}_3\text{Fe}_3\text{Cr}_1$	$\text{Al}_{17.65}\text{Co}_{35.29}\text{Fe}_{35.29}\text{Cr}_{11.76}$	Homogenized at 1573 K	127.0	1.8	143.3
		773 K-aged for 24 h	129.6	1.8	143.3
		873 K-aged for 24 h	127.8	1.9	151.2
		873 K-aged for 480 h	132.0	2.0	159.2
		973 K-aged for 24 h	129.2	3.4	270.6
		973 K-aged for 480 h	120.0	24.5	1950.2

Figure 2c–e presents the SEM images of the aged $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ alloy. After aging at 773 and 873 K for 24 h, the sizes of both macro-scaled grains and micro-scaled cells do not show significant changes for the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ alloy, which are comparable to that in the homogenized state, as shown in Figure 2c,d. With the aging temperature increasing to 973 K, the previously observed micro-scale cells disappear, as shown in Figure 2e. Moreover, it is found that cuboidal BCC nanoparticles with a size of 90 ± 12 nm are precipitated in the B2 matrix. Figure 2f displays the TEM analysis of this alloy aged at 973 K for 24 h. The TEM-DF image and the SAED pattern along the $[100]_{\text{BCC}}$ direction indicate that the coarse BCC nanoprecipitates, exhibiting a size of ~ 90 nm, are dispersed uniformly in the B2 matrix, indicating the significant microstructural evolution at 973 K.

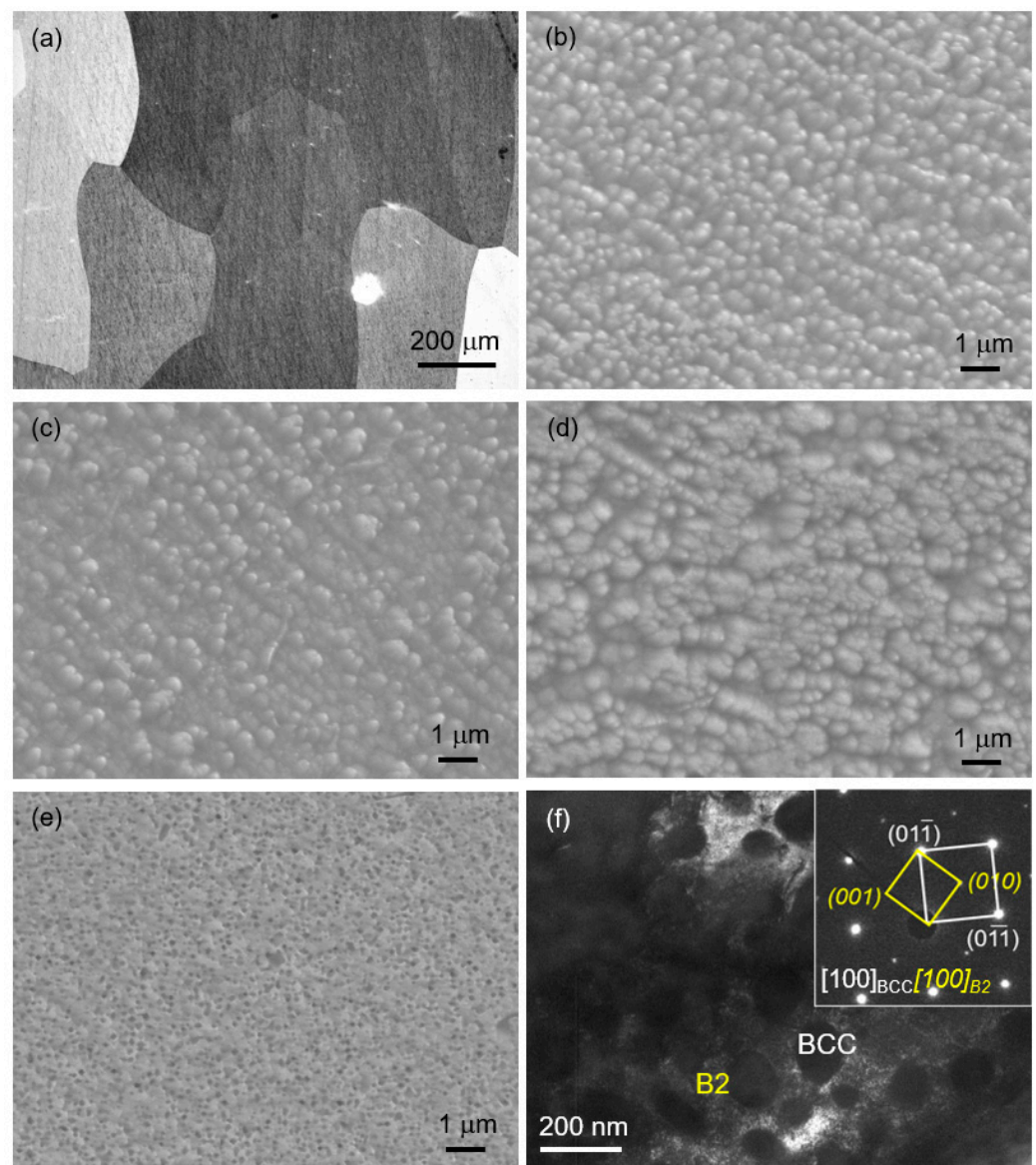


Figure 2. Microstructural characterization of the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA in the homogenized and aged states. (a) OM image in the homogenized states; (b) SEM observations in the homogenized states; (c–f) SEM observations of the alloy after aging at 773, 873, and 973 K for 24 h; (e) the TEM dark-field (DF) image and the corresponding selected-area electron diffraction (SAED) pattern of alloy after aging at 973 K for 24 h.

Figure 3a,b shows the SEM micrographs of this alloy after aging at 873 and 973 K for 480 h, respectively. It is found that in an 873 K-aged state, both macro-scaled grains and micro-scaled cells do not show significant changes, which indicates that the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ alloy exhibits the optimal thermal stability in 873 K. However, upon aging at 973 K, a significant coarsening of the BCC nanoprecipitates was found, as seen in Figure 3b. Multiple BCC nanoprecipitates are merged together during coarsening, resulting in the formation of ellipsoidal particles with a size of ~150 nm or short rod-shaped particles with a length of ~500 nm and a width of ~190 nm. Further analysis shown in Figure 3c displays the TEM examination of the alloy after aging at 973 K for 480 h. The TEM-DF image and the SAED pattern obtained along the $[110]_{\text{BCC}}$ direction also reveal that the BCC nanoprecipitates are precipitated within the B2 matrix. Furthermore, the 480 h-aged sample was characterized by STEM, and the HAADF-STEM image and corresponding TEM-EDS mapping of the BCC and B2 phases are shown in Figure 4. Obviously, the B2 matrix is

segregated in Fe and Al, while Co and Cr partition mainly within the BCC nanoprecipitates. The average compositions of BCC nanoprecipitates and B2 matrix are $\text{Al}_{5.6}\text{Co}_{20.9}\text{Fe}_{29.2}\text{Cr}_{44.3}$ and $\text{Al}_{19.8}\text{Co}_{34.4}\text{Fe}_{27.9}\text{Cr}_{17.9}$ (at. %), respectively.

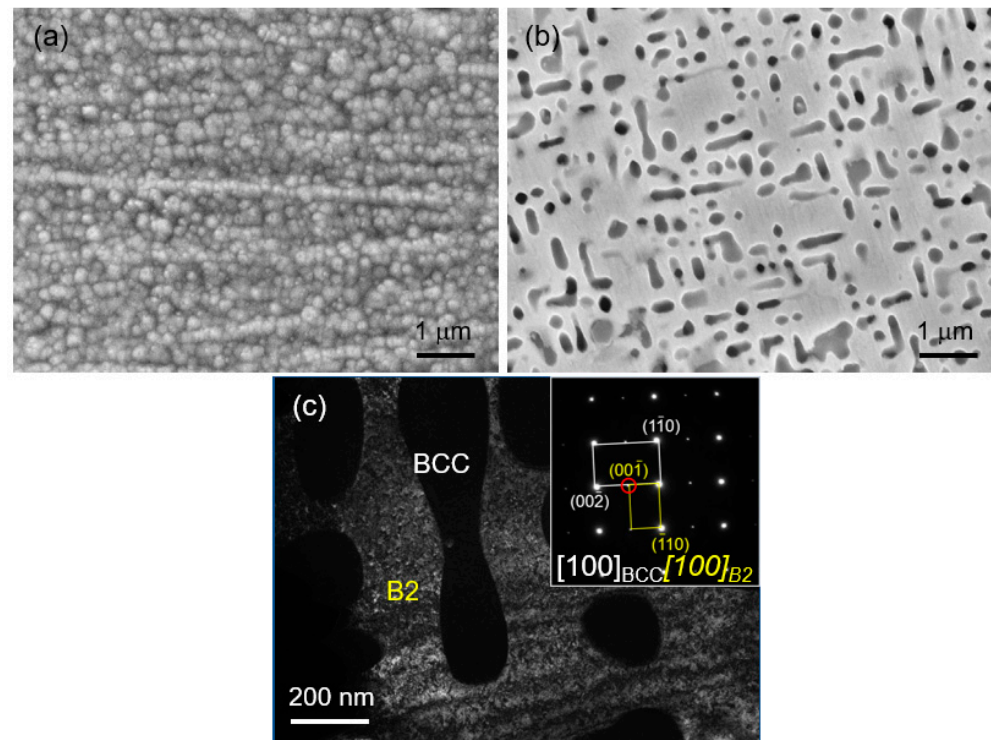


Figure 3. (a,b) SEM observations of the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ alloy after aging at 873 K (a) and 973 K (b) for 480 h respectively; (c) the TEM dark-field (DF) image and the corresponding selected-area electron diffraction (SAED) pattern of alloy after aging at 973 K for 480 h, where the diffraction spot, from which DF image are obtained, is marked by the red circle.

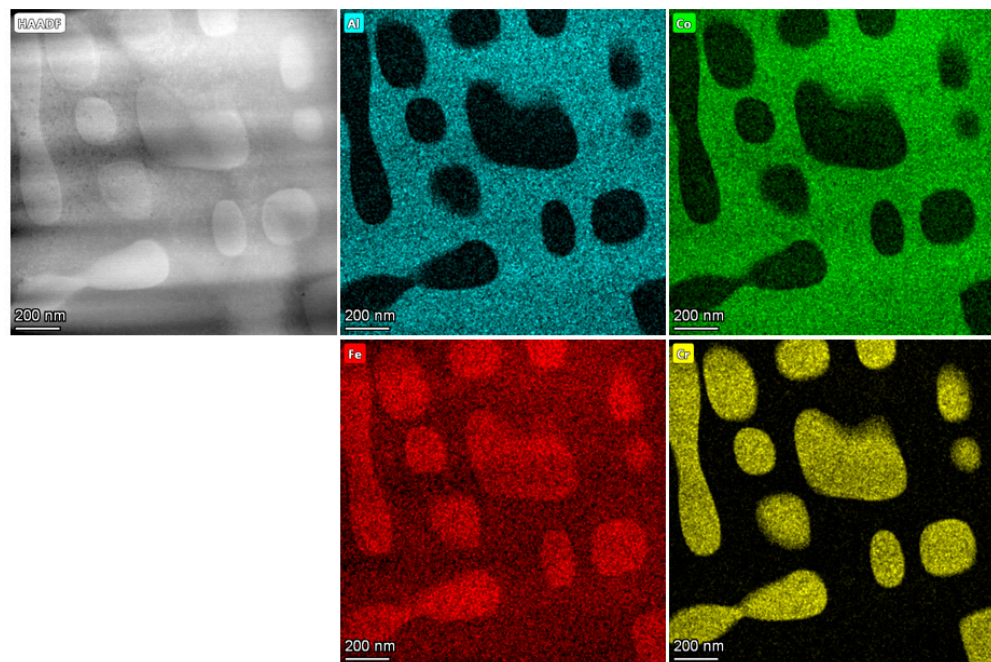


Figure 4. HAADF-STEM micrograph and corresponding EDS mapping of $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ alloy aged at 973 K for 480 h.

3.2. Soft-Magnetic Properties of $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA in Different States

The room temperature (RT) hysteresis loops of the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA, both in the homogenized and aged conditions, are displayed in Figure 5, and from these, the magnetic properties, i.e., coercivity H_C and saturation magnetization M_S , were determined, and these resulting data are given in Table 1. It can be clearly seen that this homogenized alloy demonstrated prominent soft-magnetic properties with $M_S = 127$ emu/g and $H_C = 143.3$ A/m (1.8 Oe). To further study the effect of aging temperature on the soft-magnetic properties, the hysteresis loops of the aged alloys at different temperatures are also presented in Figure 5. It is found that the M_S of the aged $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ alloy remains unmodified (~ 129 emu/g), and its coercivity increases slightly as the aging temperature increases, as demonstrated by $H_C \sim 150$ A/m (1.9 Oe) in 773~873 K-aged states and $H_C = 270\sim 374$ A/m (3.4~4.7 Oe) in 973~1073 K-aged states. Moreover, after long-term (480 h) aging at 973 K, the soft-magnetic properties of the alloy deteriorated significantly, resulting in an increase in the coercivity to $H_C = 1950.2$ A/m (24.5 Oe). In contrast, after long-term aging at 873 K, this alloy still has good soft-magnetic properties with a moderate $M_S = 132.0$ emu/g and a low $M_S = 159.2$ A/m (2.0 Oe). This would be due to the coarsening of the BCC nanoparticles, which gradually raises the coercivity to $H_C = 1950$ A/m (in the 480 h-aged state) (Figure 5 and Table 1). Consequently, the soft-magnetic properties of the 773~873 K-aged specimens are similar to those in the homogenized sample, which could imply that the microstructure in the aged state is similar to that at the homogenized state, i.e., the uniform distribution of ultrafine BCC nanoprecipitates within the B2 matrix.

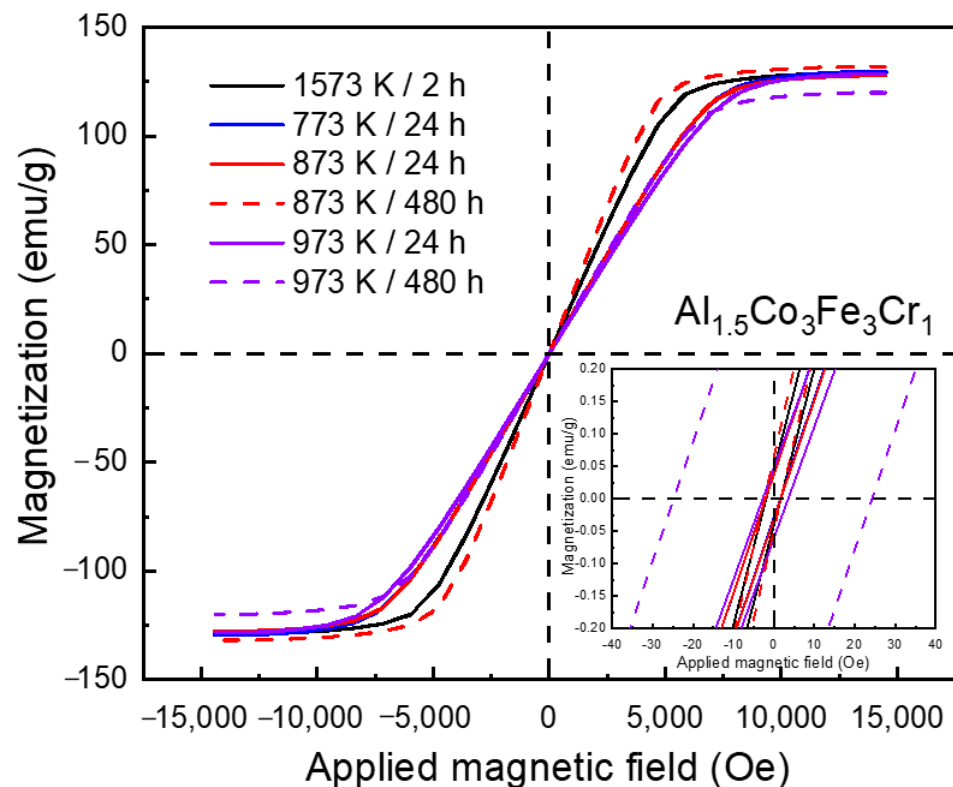


Figure 5. RT hysteresis loops of the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA in different states.

4. Conclusions

In conclusion, the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA exhibits a unique microstructure consisting of coherent BCC and B2 phases. The alloy shows excellent soft-magnetic properties in the homogenized state, characterized by high saturation magnetization ($M_S = 127$ emu/g) and low coercivity ($H_C = 143.3$ A/m). The aging temperature plays a significant role in the microstructure and magnetic properties. Aging at temperatures below 873 K has negligible effects on the microstructure and magnetic properties, indicating good thermal

stability. However, at higher temperatures (973 K) and longer aging times, the coarsening of BCC nanoprecipitates occurs, leading to a decrease in the soft-magnetic properties. The coercivity increases with aging temperature, and after long-term aging at 973 K, the alloy's soft-magnetic properties deteriorate significantly. In contrast, the alloy aged at 873 K maintains its prominent soft-magnetic properties. These results demonstrate the importance of microstructure control in optimizing the soft-magnetic properties of the $\text{Al}_{1.5}\text{Fe}_3\text{Co}_3\text{Cr}_1$ MPEA. The design of coherent microstructure provides a new development in high-performance soft magnets for high temperatures.

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Data Availability Statement: The data presented in this study are available upon request from the corresponding author. The data are not publicly available due to privacy.

Conflicts of Interest: Authors Shaoheng Sun, Yaxia Qiao, Hao Zhang, Dejun Tu and Guojun Wang were employed by the company China Electric Power Research Institute. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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