Structural and magnetic behavior of Cr$_2$Co$_{(1-x)}$Cr$_x$Al inverse Heusler alloys

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We report the structural and magnetic behavior of single phase inverse Heusler alloys Cr$_2$Co$_{(1-x)}$Cr$_x$Al ($x = 0, 0.2, 0.4$) using x-ray diffraction (XRD), Raman spectroscopy, isothermal magnetization, and magnetic susceptibility measurements. Interestingly, the Rietveld refinement of XRD data with the space group $I4/m2$ reveal a tetragonal distortion with $c/a$ ratio around 1.38 in these inverse Heusler structures. The bulk compositions have been confirmed by energy dispersive x-ray spectroscopy measurements. The active Raman mode $F_{2g}$ is observed at 320 cm$^{-1}$, which confirms the X-type Heusler structure as the A2 and B2 type structures are known to be not Raman active. The area of $F_{2g}$ mode decreases with Cr concentration, which indicates the origin of this mode due to Co vibrations. The isothermal magnetization data confirm the magnetic moment close to zero ($\leq 0.02 \mu_B/f.u.$) at $\approx 70$ kOe and negligible coercive field suggest the fully compensated ferrimagnetic nature of these samples. The susceptibility behavior indicates irreversibility between zero-field and field-cooled curves and complex magnetic interactions at low temperatures.

The full Heusler alloys are $X_2YZ$ type ternary intermetallic compounds having $X$ and $Y$ as transition metals and $Z$ as main group element or an sp-element $[1]$. In recent years many Co-based Heusler alloys (HAs) have been studied extensively due to their structural stability and half-metallic ferromagnetic nature as well as they have an advantage of high Curie temperature ($T_C$) $[2, 3]$ and also show the spin gapless semiconducting properties $[4, 5]$. In the family of CoCr based full-Heusler alloys, Co$_2$CrAl has particularly attracted huge attention due to its true half-metallic ferromagnetic (HMF) nature with $3 \mu_B/f.u.$ magnetic moment as per the band structure calculations and the Slater-Pauling rule, respectively $[6, 7]$. It is interesting to note here that Luo $et$ $al.$ studied the effect of Cr substitution on Co site and showed a transition from HMF to HM fully compensated ferrimagnetic state $[8]$. Moreover, the appreciable value of spin polarization of about 68% was also reported for Cr$_2$CoAl $[9]$. This found to be most important material as by changing the Cr concentration, we can tune the magnetic moment from $3 \mu_B/f.u.$ to zero without destroying the half-metallicity $[8]$. These compounds show the largest value of spin density among all known ferromagnetic half-metals so far. Interestingly, few Heusler alloys show half-metallic fully compensated ferrimagnetic behavior $[10]$. These materials act as half-metal as well as gapless semiconductors. In 2013, M. Meinert $et$ $al.$ theoretically investigated Cr$_2$CoAl Heusler alloys as completely compensated half-metallic ferrimagnets $[11]$. The reason for the low moment (0.01$\mu_B/f.u.$) is the internal spin compensation of the transition metals placed at different sites (Cr1: 1.36 $\mu_B$, Cr2: -1.49 $\mu_B$, Co: 0.30 $\mu_B$) and the high Curie temperature of these materials is because of their strong local moments $[11]$. Moreover, there is also possibility of showing low magnetic moments of alloy due to the anti-ferromagnetically orientation of Cr-Cr neighboring elements $[11, 12]$. The main advantage of these alloys is that they develop very low stray field or demagnetizing field, due to which they can be utilized in the fabrication of spin-torque based devices $[13]$. By calculating the lowest energy configuration, Meinert $et$ $al.$ also demonstrated that this alloy is stable as compared to their elemental constituents but not stable with respect to their binary phases thus it is easily decomposes into CoAl and Cr phases $[11]$. On the other hand, it was reported that due to the negative formation energy, Cr$_2$CoAl can possibly stabilize in the inverse (X-type) Heusler structure $[9]$.

In this paper, we report the structural and magnetic properties of single phase Cr$_2$Co$_{(1-x)}$Cr$_x$Al ($x = 0, 0.2, 0.4$) inverse Heusler alloys. The bulk compositions have been confirmed by energy dispersive x-ray spectroscopy measurements. The Rietveld refinement of XRD data space group $F43m$ confirms the X-type Heusler structure. More interestingly, our analysis with space group $I4/m2$ reveals a tetragonal distortion in these samples. Interestingly, the vibration of Raman modes with different composition have been observed. The active Raman mode $F_{2g}$ is found to be at about 320 cm$^{-1}$, which confirms the X-type Heusler structure as the A2 and B2 type structures are known to be not Raman active. The area of $F_{2g}$ mode decreases with Cr concentration, which indicate the origin of this mode due to Co vibrations. The magnetic analysis confirms zero moment confirming fully compensated ferrimagnetic nature and complex interactions at low temperatures.

The polycrystalline samples of Cr$_2$Co$_{(1-x)}$Cr$_x$Al ($x = 0, 0.2, 0.4$) were prepared by arc melting from CRESTOR Vacuum Industries, USA. In order to reduce the oxygen partial pressure, the chamber was evacuated and flushed with high purity argon. This process was repeated few times and a small piece of Ti metal is melted (which act as getter pump of oxygen) before melting the sample. The appropriate quantities of the constituent el-
emergencies of 99.99% purity (from Sigma-Aldrich) were arc melted under an inert argon atmosphere. The ingot was flipped and melted 4-5 times to ensure the homogeneity. For further homogenization and larger grain size samples, the ingot materials were wrapped in Mo foils and sealed in evacuated quartz ampules and then annealed at 1173 K for five days in high temperature box furnace from Nabertherm, GmbH, Germany. The samples were finally quenched in ice water to obtain the highest degree of chemically ordered phase. The bulk compositions have been confirmed by energy dispersive x-ray spectroscopy measurements. We use x-ray diffraction (XRD) with Cu Kα (λ = 1.5406 Å) radiation for structural study and analyzed the XRD data by Rietveld refinement using FULLPROF package where the background was fitted using linear interpolation between the data points. The Raman measurements were carried out at room temperature using a LabRAM HR evolution Horiba spectrometer. A He-Cd laser with 325 nm excitation wavelength, 1200 lines per mm grating and 1 mW laser power was used. The magnetic measurements are performed using VSM mode in a physical properties measurements system (PPMS EVERCOOL-II) from Quantum design.

In $X_2YZ$ type full-Heusler alloys, the unit cell consists of four interpenetrating fcc sublattices with the positions A (0 0 0), B (0.25, 0.25, 0.25), C (0.5, 0.5, 0.5) and D (0.75, 0.75, 0.75). The perfectly ordered $L2_1$-type structure, with space group $Fnm3m$, consists X atoms at A and C sites, Y atoms at B site and Z atoms at D site. If we see diagonally from any atom then the sequence of the sites is found to be as $X - Y - X - Z$. In $X_2YZ$ compounds, if the atomic number of Y element is higher than the X element (as the case in the present study) an inverse Heusler structure (also called X-type structure with $F43m$ space group) is observed [15]. In this structure half of the X atoms are replaced by Y atoms so the position of the atoms changes as $X - X - Y - Z$, i.e., the difference between the position of two X atoms in particular direction is $\frac{1}{2}$ and $\frac{1}{2}$ in regular and inverse Heusler structures, respectively [1].

In Figs. 1(c–f), we present the room temperature XRD and inverse Heusler structures, respectively [1, 14, 15]. The Rietveld refinement of the XRD pattern with cubic space group $F\bar{4}3m$ which indicates the possibility of two different phases, fitted with the space group $I\bar{4}m2$, is shown in the Figs. 1(e, f) and the respective insets are the enlarged view of most intense peak for the sake of clarity of tetragonal nature. XRD patterns (open circles) and Rietveld refinement (black line) of $Cr_2Co_{1-x}Cr_x Al (x = 0, 0.2, 0.4)$ with difference profile (blue line) and Bragg peak positions (short vertical bars, green).

The lattice parameters of $Cr_2Co_{1-x}Cr_x Al$ in the tetragonal phase with $I\bar{4}m2$ space group is summarized in Table 1 for all the samples in inverse tetragonal phase. The Wyckoff position of the atoms of $Cr_2CoAl$ alloy in the inverse tetragonal structure; Cr atoms occupy two different sites as Cr1 at 2b (0, 0, 0.5) and Cr2 at 2c (0, 0.5, 0.25). The Co and Al atoms occupy at site 2d (0, 0.5, 0.75) and 2a (0, 0, 0) respectively [12]. In order to find a best fit, we had to include the disorder between Cr and Al [18] [19], which found to be about Cr1/Al (55%/45%) and Cr2/Al (45%/55%) at 2b and 2a sites, respectively for all the samples in inverse tetragonal phase. The lattice constant values obtained from the XRD patterns and tetragonality ratio (c/a) are summarized in Table I for all three samples, which agree with the reported values for $Cr_2CoGa$ in ref. [18]. We found an increment in the lattice parameters with the substitution of Cr at Co site, which is expected as the atomic radius of the Cr (128 pm)
is larger than that of the Co (125 pm).

In Fig. 2, we show the Raman spectroscopy data of Cr$_2$Co$_{1-x}$Cr$_x$Al ($x = 0, 0.2, 0.4$) measured with 325 nm excitation wavelength at room temperature. In general, it is not easy to observe the Raman signal from the metallic samples; however, it is interesting to note that Zayak et al. predicted about the experimental evidence of Raman vibrational modes in metallic Heusler alloys [20]. The authors show that for the Heusler alloys, the optical modes are splitted into three well separated triply degenerate modes ($F_{2g} + 2F_{1u}$) where $F_{2g}$ has been found to be Raman active, whereas two $F_{1u}$ modes are IR active [20]. Interestingly, we observed the most intense Raman mode ($F_{2g}$) at wavenumber $\approx 320$ cm$^{-1}$, which is in agreement with the value reported in the literature [21,22]. It should be noted that Zhan et al. used Raman scattering to study Co$_2$FeAl Heusler compound across the Curie temperature and observed that the intensity of Raman signal strongly depends on Co atoms vibrational modes. In the present study, we found that the intensity of $F_{2g}$ peak is decreasing with decrease in the amount of Co, which indicates the origin of this mode due to the vibrations of Co atoms in the lattice. We have fitted the $F_{2g}$ peak for all three samples with Gaussian function and then plotted the area as a function of Cr concentration, as shown in the inset of Fig. 2. This clearly indicates the decrease in the Raman signal with Cr concentration.

Also according to the literature [20,21] the existence of Raman modes indicates the presence of X-type structure because A2 and B2 type structures are not Raman active. We have also observed two broad modes at around 800 cm$^{-1}$ and 1025 cm$^{-1}$, which are consistent with reported in ref. [23] on the similar Heusler alloys.

Further, in order to understand the magnetic properties and moment of Cr$_2$CoAl we have measured isothermal magnetization versus magnetic field (M–H) and magnetic susceptibility $\chi$ versus temperature (M–T). Figs. 3(a, b) show the M–H curves of Cr$_2$Co$_{1-x}$Cr$_x$Al ($x = 0, 0.4$) measured at 300 K and 50 K, respectively, which clearly indicate a very small moment of the order of $10^{-3}$ $\mu_B$/f.u. and non saturating behavior of magnetization up to $\pm$ 50 kOe for both the samples. It has been reported that the formation of impure CoAl phase, which is paramagnetic in nature, can be responsible for the linear behavior in the M–H curves [11]. However, the absence of this CoAl phase in the XRD data of our samples rule out this possibility. Our results suggest an antiferromagnetic coupling between the Cr–Cr neighboring atoms, consistent with ref. [9]. On the other hand, at 300 K, a small finite hysteresis has been observed for the $x = 0$ sample (50 Oe), but not for the $x = 0.4$ sample, as clearly seen in the zoomed view in the inset of Fig. 3(a). Also, at 50 K, the values of coercivity were found to be 200 Oe and 100 Oe for the $x = 0$ and 0.4
samples, respectively [inset of Fig. 3(b)], which suggests presence of ferromagnetic order. It has been shown that the partial density of states for minority (majority) spins are located above (below) the Fermi level for Cr1 whereas for Cr2 it is reversed, which implies their spin moments in antiparallel configuration [15]. The observed magnetic behavior in Figs. 3(a, b) is consistent as the Co at Y site couples ferromagnetically with the nearest neighbor Cr and antiferromagnetically with second nearest neighbor Cr and the two distinct Cr sites couple ferrimagnetically [17]. This type of alignment is attributed due to a competition between the intra-atomic exchange splitting of the magnetic atom d states and the inter-atomic covalent interaction of d states from atoms at different sites [24]. This also agrees with the theoretical predication of half-metallic fully compensated ferrimagnetic nature in the Cr2CoAl sample [11, 12].

In Figs. 3(c, d), we show the magnetic susceptibility \( \chi \) as a function of temperature in both zero-field-cooled (ZFC) and field-cooled (FC) modes for the \( x = 0 \) and 0.4 samples, respectively measured in the temperature range of 10–380 K and at 100 Oe magnetic field. For the \( x = 0 \) sample, the moment is fairly constant and close to zero, i.e., the sample is in fully compensated ferrimagnetic state above 200 K, which is defined a compensation temperature, as marked in the derivative shown in the inset of Fig. 3(c). Interestingly, the moment increases significantly below \( \sim 200 \) K and the irreversible behavior between FC and ZFC moments is clearly evident. Furthermore, the ZFC curve shows a down hump \( \sim 50 \) K and below around 25 K there is a sharp increase in the moment for both FC and ZFC modes. The behavior of temperature dependent magnetization for the \( x = 0 \) sample is in good agreement with the only reported in ref. [16]. On the other hand, it is important to note here that Jamer et al. attributed the low temperature behavior as paramagnetic due to the presence of extra CoAl phase in their sample [16], which clearly is not the case in the present study as evident in our XRD analysis shown in Fig. 1. With increasing Cr concentration at Co site, i.e., for the \( x = 0.4 \) sample in Fig. 3(d), the compensation temperature shifted to around 220 K, which is clearly visible in the derivative plot as shown in the inset. Below this temperature, we observe bifurcation in ZFC and FC curves and consistently increase in the moment till lowest measured temperature. This magnetization behavior for both the samples suggests for a complex magnetic interactions at low temperatures, which motivates to study these materials using neutron diffraction for further understanding these interactions and the role/possibility of atomic disorder [2]. Though the Curie temperature (\( T_C \)) is expected at around 700 K [16], the magnetization measurement at high temperature would require to find exact value of \( T_C \) of these materials [25]. Interestingly, the nature of competing magnetic behavior is clear from the irreversibility in ZFC–FC curves where the fully compensated feature is very sensitive to the compositions [26].

Note that the half-metallic full-Heusler alloys follow a Slater-Pauling behavior, i.e., the total spin magnetic moment per formula unit, \( M_t \), in \( \mu_B \) scales with the total number of valence electrons, \( Z_t \), following the rule: \( M_t = Z_t - 24 \) [15]. In the case of fully compensated ferrimagnet (FCF), the total spin magnetic moment should be zero like \( \text{Cr}_2\text{CoGa} \) and \( \text{Fe}_2\text{VGa} \) having exactly 24 valence electrons and the ground state of \( \text{Cr}_2\text{CoAl} \) is found to be ferrimagnetic in inverse Heusler structure [11]. However, the ferrimagnetic state of the \( \text{Cr}_2\text{CoAl} \) sample arises due to the antiferromagnetic coupling of Cr-Cr atoms of inequivalent nearest neighbors and show the nature of fully compensated ferrimagnetic [11]. In a more precise way, for these type of alloys, there is a competition between the magnetic states of atoms, which decides whether the alignment of the moments should be ferromagnetic or antiferromagnetic. Therefore, \( \text{Cr}_2\text{CoAl} \) possess almost vanishing total spin magnetic moments in FCF states because of the antiferromagnetic alignment of the Cr-Cr inequivalent nearest neighbouring atoms due to the direct interaction between d states [9]. Moreover, the Curie temperature of these alloys is expected to be high [27], which makes them most promising candidate for devices.

In summary, we successfully prepared single phase \( \text{Cr}_x\text{Co}(1-x)\text{Cr}_z\text{Al} \) \( (x = 0, 0.2, 0.4) \) and investigated the structural and magnetic behavior using x-ray diffraction (XRD), Raman spectroscopy, isothermal magnetization, and magnetic dc susceptibility measurements. The Rietveld refinement of XRD patterns confirm the X-type Heusler structure, and interestingly a tetragonal distortion (space group \( \text{I}4\text{m}2 \)) has been observed in these samples where the \( c/a \) value is found to be around 1.38. The active Raman mode \( F_{2g} \) is found to be at about 320 cm\(^{-1} \), which confirms the X-type Heusler structure as the A2 and B2 type structures are known to be not Raman active. The area of \( F_{2g} \) mode decreases with Cr concentration, which suggests the origin of this mode due to Co vibrations. The susceptibility data show irreversible behavior between ZFC and FC curves, which indicate complex magnetic interactions. The magnetization data show that the magnetic moment is close to zero at around 70 kOe, which confirms the fully compensated ferrimagnetic nature of these inverse Heusler alloys.

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