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Abstract

Single crystals of PrCo₂ have been grown from flux starting from Pr_{0.44}Co_{0.56} that was synthesized by arc melting of stoichiometric mixtures of the elements. The Pr_{0.44}Co_{0.56} ingot was loaded in a Boron Nitride (BN) crucible and sealed inside a quartz tube under vacuum. The quartz ampoule was heated to 1203 K, and then slowly cooled down to 923 K at a rate of 2 K/h. The obtained crystals have octahedral shapes and are of cubic C15 Laves phase structure confirmed by room temperature powder and single-crystal x-ray diffraction measurements. Magnetic susceptibility data show the Curie temperature $T_C = 46.7$ K. The experimental value of saturation magnetization $M_s = 3.2 \mu\text{B}/\text{f.u}$ is consistent with our theoretical calculations.

Keywords

A2. Growth from melt, A2. Single crystal growth, B1. Rare earth compounds, B2 Magnetic materials

Disciplines

Inorganic Chemistry | Materials Chemistry | Metallurgy

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Growth of PrCo₂ single crystals with a Boron Nitride crucible

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Single crystals of PrCo₂ have been grown from flux starting from Pr_{0.44}Co_{0.56} that was synthesized by arc melting of stoichiometric mixtures of the elements. The Pr_{0.44}Co_{0.56} ingot was loaded in a Boron Nitride (BN) crucible and sealed inside a quartz tube under vacuum. The quartz ampoule was heated to 1203 K, and then slowly cooled down to 923 K at a rate of 2 K/h. The obtained crystals have octahedral shapes and are of cubic C15 Laves phase structure confirmed by room temperature powder and single-crystal x-ray diffraction measurements. Magnetic susceptibility data show the Curie temperature $T_C = 46.7$ K. The experimental value of saturation magnetization $M_s = 3.2 \mu_B/\text{f.u}$ is consistent with our theoretical calculations.

Key words: A2. Growth from melt, A2. Single crystal growth, B1. Rare earth compounds, B2
Magnetic materials

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1. Introduction

Complex magnetism that results from varying exchange and crystal-field interactions between the lanthanide and Co sublattices in intermetallic $R\text{Co}_2$, where R = rare-earth, has been extensively studied [1,2]. The magnetic interactions consist of two distinct contributions: (i) local $4f-5d$ exchange, which is linearly dependent on the rare-earths' de Gennes factor; (ii) $5d-3d$ short range exchange interactions, through hybridization effects, which is proportional to the number of neighbouring $3d$ atoms to an R site and their moments [3]. Among $R\text{Co}_2$ compounds, ErCo_2 , HoCo_2 and DyCo_2 undergo first-order phase transition at the respective Curie temperatures, T_C , whereas TbCo_2 , GdCo_2 , SmCo_2 , NdCo_2 , and PrCo_2 order magnetically via second-order transformations [4]. The nature of the phase transitions at T_C in PrCo_2 and NdCo_2 has been the subject of a recent discussion [5-8] based on experiments performed on polycrystalline materials. Because $R\text{Co}_2$ compounds form peritectically [9], a long-time annealing below the corresponding peritectic reaction temperatures (e.g., 1210 K for PrCo_2) is required to obtain phase-pure, stoichiometric samples. Single crystals are, therefore, crucial to determine intrinsic physical properties of the $R\text{Co}_2$ compounds, in particular the anisotropic magnetism. In the past, a few groups have reported the growth of $R\text{Co}_2$ single crystals. For example, a single crystal of CeCo_2 was successfully grown by zone melting starting from $\text{CeCo}_{1.7}$ composition by zone melting [10]. The obtained ingot was about 10 mm in diameter and 20 mm in length, and consisted of several large grains. In this work we report growth of high quality PrCo_2 single crystals by using a self-flux method in a Boron Nitride (BN) crucible. Results described below should be applicable to grow other $R\text{Co}_2$ single crystals with minor adjustments.

2. Experimental

The Pr and Co metals were weighed in a molar ratio Pr:Co = 0.44:0.56 (i.e. PrCo_{1.27}) in a glovebox. This stoichiometry was projected suitable for flux growth, as determined from the published Pr-Co binary phase diagram [9]. The PrCo₂ phase of interest precipitates within the temperature range of 861 < T < 1210 K while the Pr concentration varies from 44 to 60 at.% [9]. By choosing the starting Pr:Co = 0.44:0.56 molar ratio, one may expect high yield of large single crystals while slowly cooling across such a wide temperature range. The metals were mixed and pre-reacted by using arc melting on a water-cooled copper hearth in an argon atmosphere. The arc-melted Pr_{0.44}Co_{0.56} alloy was then loaded in alumina and BN crucibles and sealed in quartz tubes. The quartz ampoules were heated to 1203 K, i.e. to just above the liquidus temperature, and then cooled down to 923 K at a rate of 2 K/h, after which the material was allowed to cool quickly (200 K/h) to room temperature. Powder XRD measurement was conducted for phase identification with the help of a PANalytical x-ray diffractometer using Cu $K\alpha$ radiation. Crystal structures were established by single crystal x-ray diffraction (XRD) using a Bruker SMART APEX II diffractometer with Mo $K\alpha$ radiation. Magnetization measurements were performed by using Physical Property Measurement System (PPMS, Quantum Design) equipped with Vibrating Sample Magnetometer (VSM). The magnetization data were collected on heating after cooling the sample to the desired temperature under zero magnetic field and during cooling in magnetic field, henceforth ZFC and FC protocols, respectively.

3. Results and discussion

3.1 Flux growth in an alumina crucible

In Fig. 1, we show the photographs of single crystals grown with the alumina crucible. As visible in Fig. 1a, a shell forms on the surface of the ingot in contact with the crucible. The formed single crystals are plate-like, exhibiting hexagonal shape symmetry, as illustrated in Fig. 1b. Composition analysis by using Energy-Dispersive X-Ray Spectroscopy (EDS) shows that crystals contain aluminum, and the crystal analyzed had chemical composition of $\text{PrCo}_{2.74}\text{Al}_{0.26}$. Single crystal diffraction measurements reveal a hexagonal structure with the space group $P6_3/mmc$; See details in Tables 1 and 2. The lattice parameters are $a = 5.16280(10)$ Å and $c = 16.4686(3)$ Å, and are similar to the known ternary compound $\text{Pr}_3\text{Co}_8\text{Al}$ ($\text{PrCo}_{2.67}\text{Al}_{0.33}$) [11]. Clearly, the Pr-Co liquid reacts with the alumina crucible, whereas Pr reduces metallic aluminum from its oxide even at the relatively low temperature of 1203 K when held for a long time, according to the reaction $2\text{Pr} + \text{Al}_2\text{O}_3 = 2\text{Al} + \text{Pr}_2\text{O}_3$.

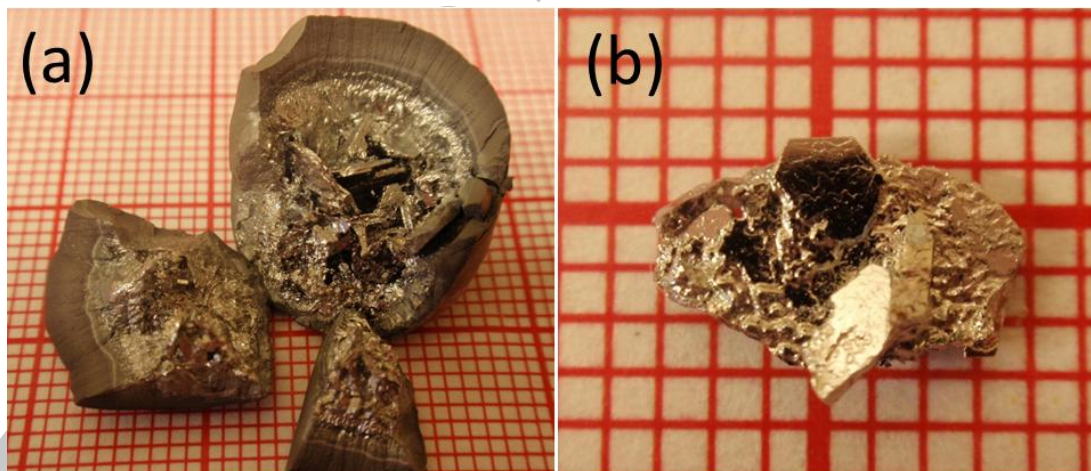


Figure 1. (a) A shell formed on the surface of the ingot in contact with the crucible material with some single crystals formed inside. (b) Enlarged view of a plate-like hexagonal single crystal formed inside the ingot on a square graph paper with 1 mm grid.

3.2 Flux growth in a BN crucible

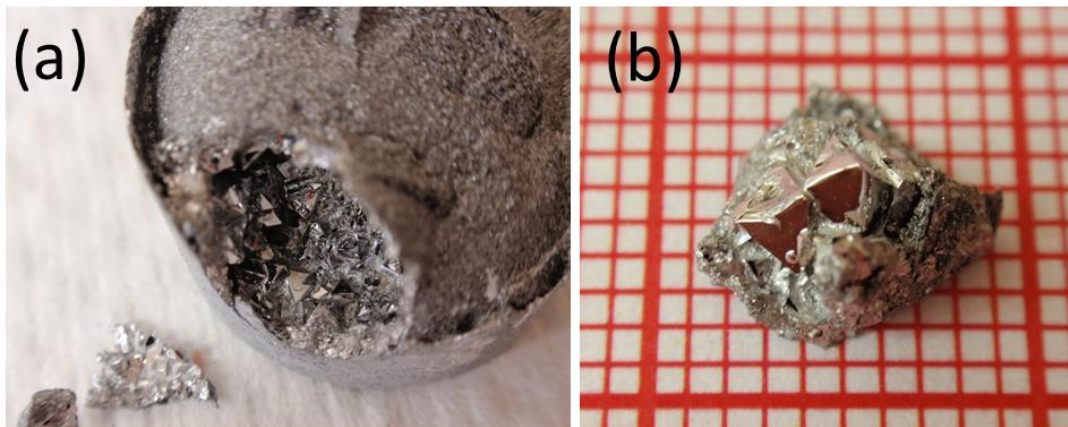


Figure 2. (a) Top view of PrCo_2 crystals grown from flux in a BN crucible. (b) Enlarged view of two octahedral crystals on a square graph paper with 1 mm grid.

Because Pr-Co liquid reacts with alumina crucible, we choose a more inert BN crucible to attempt growth of PrCo_2 crystals. Figure 2(a) shows that the upper part of the ingot is hollow. After breaking the upper crust, numerous crystals are found inside. Figure 2(b) shows two octahedral crystals, about 2 mm along the edge weighing ~4 mg each. The octahedral crystals are further identified as the cubic structure with space group $Fd\bar{3}m$ through single crystal diffraction measurements; See details in Tables 1 and 3. The lattice parameter $a = 7.3052(4)$ Å matches the lattice parameter of PrCo_2 compound reported in the literature [12].

Table 1. Crystal structure data and refinement results of $\text{PrCo}_{2.74}\text{Al}_{0.26}$ and PrCo_2 single crystals. The data were collected at 296 K on Bruker SMART APEX II diffractometer with Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å).

Chemical formula	$\text{PrCo}_{2.74}\text{Al}_{0.26}$	PrCo_2
Space group	$P6_3/mmc$	$Fd\bar{3}m$

a (Å)	5.1628(1)	7.3052(4)
b (Å)	5.1628(1)	
c (Å)	16.4686(3)	
Z	6	8
Refinement method	Full-matrix least-squares on F^2	
Goodness-of-fit on F^2	1.075	1.219
R indices	R1 = 0.0271, wR2 = 0.0687	R1 = 0.0171, wR2 = 0.0314

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for $\text{PrCo}_{2.74}\text{Al}_{0.26}$ single crystals.

Atoms*	x	y	z	U(eq)
Pr1	2/3	1/3	0.4610(1)	14(1)
Pr2	2/3	1/3	1/4	17(1)
Co1	0.1657(1)	0.3313(2)	0.3713(1)	13(1)
Co2	0	0	1/2	14(1)
M1	0	0	1/4	20(1)
M2	1/3	2/3	1/4	20(1)

*M1 = 0.48 (2) Co + 0.52(2) Al; M2 = 0.73(2) Co + 0.27 (2) Al

Table 3. Atomic coordinates and equivalent isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for PrCo_2 single crystals.

	x	y	z	U(eq)
Pr	3/8	3/8	3/8	11(1)
Co	0	0	0	9(1)

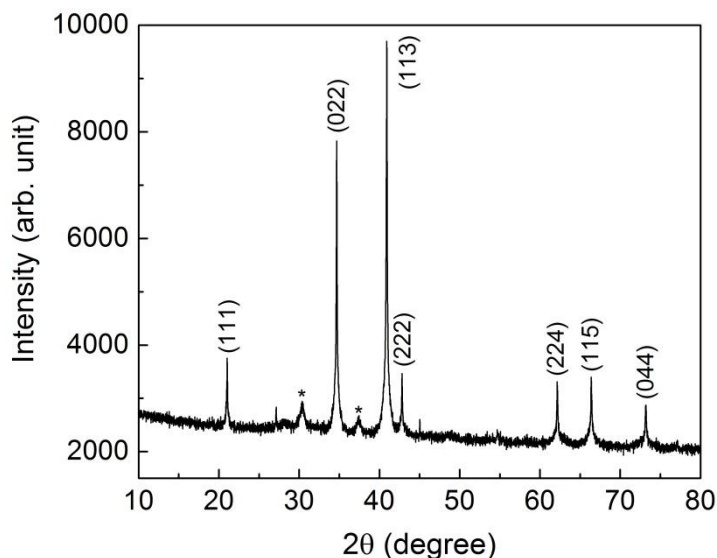


Figure 3. Powder XRD pattern of crushed PrCo_2 single crystals. Asterisks indicate the impurity phase.

In order to check phase purity of PrCo_2 single crystals, we ground as-grown single crystals into fine powder and performed powder XRD measurements, as shown in Fig. 3. One can see that the main diffraction peaks can be indexed with the space group $Fd\bar{3}m$, consistent with the single-crystal diffraction result. The impurity phase was detected, as indicated by asterisks. It is identified to be Pr_3Co [13], which forms via a congruent melting below 852 K in the phase diagram [9].

To check the magnetic transition of single crystals, we have performed magnetization measurements on the obtained crystals. It is difficult to precisely align the small octahedral crystals with the applied field. The orientation of the crystal is random with respect to the field direction. Figure 4 shows the magnetization as a function of temperature. One magnetic transition occurs at $T_C = 46.7$ K, similar to the reported Curie temperature 49-50 K in PrCo_2 polycrystals [12,14,15]. Negative magnetization observed below 20 K in ZFC data measured upon heating in low fields (less than 500 Oe) shown in Fig. 4a could be caused by the remnant field in PPMS superconducting magnet. Figure 4b shows that the magnetic transition broadens with increasing magnetic fields.

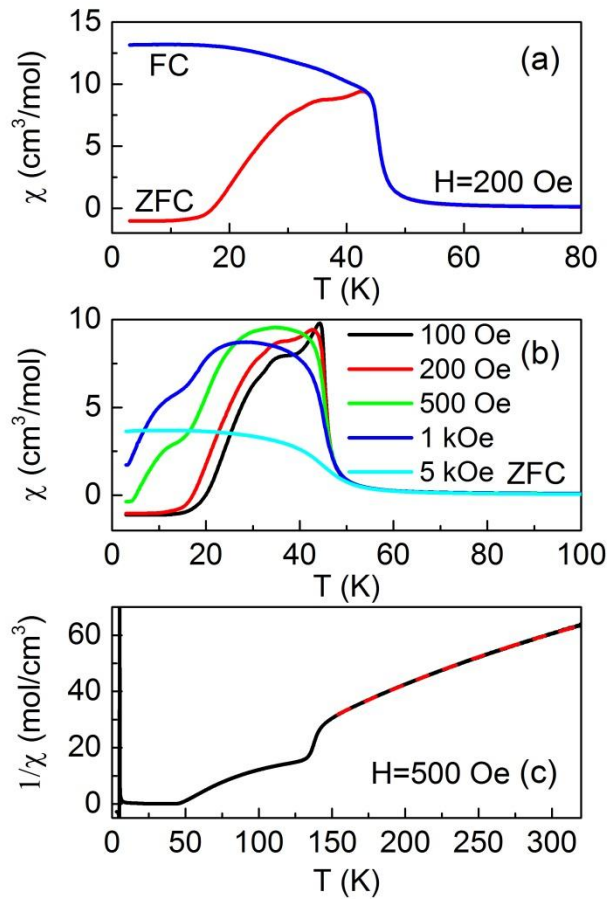


Figure 4. (a) Temperature dependence of magnetization of PrCo₂ crystals measured at $H=200$ Oe. (b) Temperature dependence of magnetization measured at $H=100, 200, 500, 1000,$ and 5000 Oe. (c) The Curie-Weiss law fitting to the high temperature part of the magnetization data, as shown by red dashed line. The orientation of the crystal is random in the measurements.

Figure 4c shows the temperature dependence of magnetic susceptibility $\chi(T)$ fitted by a modified Curie-Weiss law $\chi = \frac{C}{T-\theta_p} + \chi_0$, where C , θ_p , and χ_0 are the Curie constant, the Weiss temperature, and the temperature-independent magnetic susceptibility, respectively. The Weiss temperature θ_p of 52.2 K and the effective magnetic moment $p_{\text{eff}}=4.52 \mu_B$ are consistent with those reported for polycrystalline samples [16]. A kink is observed at $T\sim 133$ K. The single crystals were manually separated from the matrix. Although most of flux was removed under the microscopy, the remaining impurity phase should be responsible for the occurrence of such kink. We also notice that a low-temperature transition occurs at $T\sim 13$ K under the fields of 500 and 1000 Oe in Fig. 4b, which is consistent with the T_C of 14 K for Pr₃Co compound [17]. Compare with the effective moment of PrAl₂ which is $3.5 \mu_B$ in the paramagnetic region while Al is nonmagnetic [18], the $p_{\text{eff}} = 4.52 \mu_B$ for PrCo₂ is coming from both Pr and Co. The effective moment of Co in PrCo₂ crystals estimated by subtracting the Pr contribution [p_{eff} (theoretical) = $3.58 \mu_B$] is found to be $1 \mu_B$.

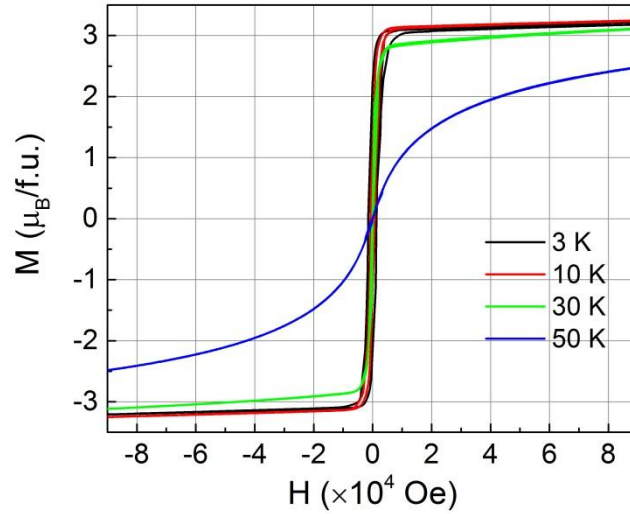


Figure 5. Magnetic hysteresis loops of PrCo₂ crystals measured at $T=3, 10, 30$ and 50 K.

Figure 5 shows the magnetic hysteresis loops of PrCo₂ crystals measured at $T = 3, 10, 30,$ and 50 K. A clear hysteresis behavior is observed below $T_C = 46.7$ K. At $T = 50$ K, slightly above T_C , hysteresis behavior almost disappears. At $T = 3$ K, the coercive field H_c is 1.3 kOe while magnetization saturates above 5 kOe. The saturation magnetization $M_s = 3.2 \mu_B/\text{f.u.}$ at $T = 3$ K and $H = 90$ kOe. A powder neutron diffraction study of PrCo₂ compound yielded Pr and Co moments of $2.9 \mu_B$ and $0.3 \mu_B$, respectively [19]. The saturation moment in the present study is very close to the ordered moment revealed by powder neutron diffraction measurements.

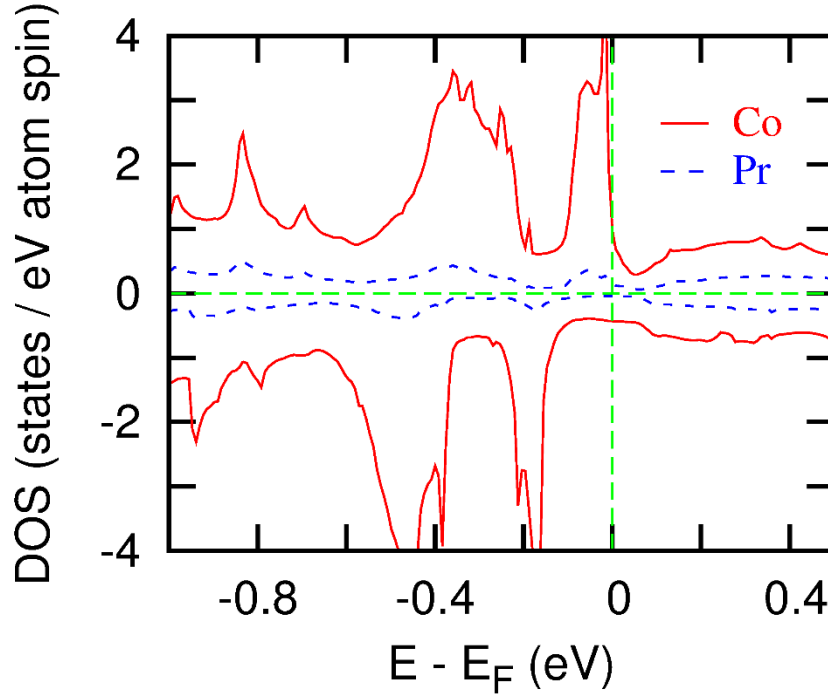


Figure 6. Partial density of states of Co and Pr atoms in PrCo_2 .

In order to understand the underlying mechanism involved in the magnetism and validate key characterization results obtained from the grown single crystals of PrCo_2 , we have also performed density functional theory (DFT) calculations by employing local spin density approximation (LSDA) including onsite electron correlation and spin orbit coupling in conjunction with full-potential linearized augmented plane wave (FP-LAPW) method [20, 21]. The theoretical calculations predict net magnetic moment of $3.11 \mu_B/\text{f.u.}$, which agrees with the experimental value of $3.2 \mu_B/\text{f.u.}$ Here, Pr $4f$ orbital magnetic moments and Co $3d$ spin magnetic moments form parallel alignment, where Pr $4f$ spin moments and Co $3d$ spin form antiparallel alignment. We would like to mention some earlier calculated results on PrCo_2 . The total moment was calculated as 1.15 and $1.05 \mu_B/\text{f.u.}$ by the full-potential non orthogonal local-orbital minimum basis method (FPLO) using LSDA and generalized gradient approximations (GGA), respectively [22]. Bakkari *et. al.* reported the total moment of $4.55 \mu_B/\text{f.u.}$ by using the FP-

LAPW method in a GGA + U approximation [23]. In both cases the orbital moments are not clearly mentioned. However, we have precisely calculated both spin and orbital moments and our calculated net moment agrees well with the experimental value. Figure 6 shows partial density of states (DOS) for Co atoms in PrCo₂. There is a clear exchange splitting between spin up and spin down states just below the Fermi level indicating an expected itinerant magnetic behavior exhibited by Co atoms in PrCo₂. The occupied Pr 5*d* states in the valence band hybridize with the Co 3*d* states. Further the Pr 4*f* states located around ~ -3 eV below the Fermi level (not shown) overlap with the hybridized Pr 5*d* and Co 3*d* states leading to strong 4*f*-5*d*-3*d* exchange. This exchange is responsible for the indirect 4*f*-4*f* exchange that gives rise to long range ferrimagnetism in PrCo₂. The sharp DOS peak below the Fermi level also indicates a possibility of structural distortion (instability) of cubic structure as indicated experimentally earlier by Gratz *et al* [24]. The total energy calculations confirm that the distorted phase along the [001] direction is indeed stable over the cubic phase.

4. Conclusions

We find crucible materials are crucial to grow PrCo₂ single crystals by using self-flux method. In this study, BN crucible stands out in the growth. It should be pointed out that the key to a successful growth of rare-earth transition-metal intermetallics is to avoid using alumina crucibles rather than using BN crucibles. The other options such as zirconia, tantalum, and tungsten crucibles should be considered as possible replacement of BN crucible in the growth. Our growth protocol is instructive for the crystal growth of other RCo₂ compounds. The obtained crystals have an octahedral shape and show the Curie temperature at $T_C = 46.7$ K. Theoretical calculations confirm the experimentally observed saturation moment $M_s = 3.2 \mu_B/\text{f.u.}$

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Highlights

- PrCo₂ single crystals grown with a Boron Nitride (BN) crucible
- Reaction between the Pr-Co liquid melt and alumina crucible
- The experimentally observed saturation moment matches theoretically calculated value

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