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Phase diagram of R-Fe-Co pseudoternary system with $R \leq 33.3\%$ (mole fraction, $R = \text{Sm}_{0.5}\text{Dy}_{0.5}$)^①

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[Abstract] Phase equilibria of the R-Fe-Co pseudoternary system with $R \leq 33.3\%$ (mole fraction, $R = \text{Sm}_{0.5}\text{Dy}_{0.5}$) were determined in an isothermal section at 1 073 K and a vertical section of RFe_2RCO_2 by using OM, X-ray diffractometer, EPMA DTA techniques. There are seven intermetallic phases: $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_2$, $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_3$, $(\text{Sm}, \text{Dy})_6(\text{Fe}, \text{Co})_{23}$, $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_7$, $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_5$, $\text{Th}_2\text{Ni}_{17}$ type and $\text{Th}_2\text{Zn}_{17}$ type $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_{17}$. The $(\text{Sm}, \text{Dy})_6(\text{Fe}, \text{Co})_{23}$ phase dissolves 36% Co (mole fraction) at 1 073 K. However, the $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_7$ phase in $\text{R}_2(\text{Fe}_{1-x}\text{Co}_x)_7$ alloys dissolves about 19% Fe (mole fraction) at 1 073 K.

[Key words] rare earth-iron-cobalt; phase diagram; structure; compounds

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1 INTRODUCTION

The two successful families of rare-earth cobalt permanent magnets produced commercially are SmCo_5 and $\text{Sm}_2\text{Co}_{17}$. The SmCo_5 magnets have been produced with very high intrinsic coercivity and the $\text{Sm}_2\text{Co}_{17}$ magnets have the low temperature coefficient of remanence ($-0.12\% \cdot ^\circ\text{C}$)^[1]. In recent years, the calculation of magnetic properties of $\text{SmCo}_5/\text{Co}_{1-x}\text{Fe}_x$ composite multilayer has found that its energy product is about 520 kJ/m^3 (65 MGOe), which is almost twice the value for the hard phase^[2]. The investigation of the structure and magnetostriction for $(\text{Sm}, \text{Pr})(\text{Fe}, \text{Co})_2$ ^[3], $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_2$ ^[4] and $(\text{Dy}, \text{Pr})(\text{Fe}, \text{Co})_2$ ^[5] alloys has found that substitution of Co for Fe can increase the Curie temperature and saturated magnetostriction, λ_{11} ^[3] and λ_{00} ^[4]. At the same time, addition of Dy to SmCo_5 compound causes the temperature coefficient of remanence to be decreased^[6]. In order to develop permanent magnetic rare earth-iron alloys and magnetostrictive materials, an investigation of the effect of partial substitution of Dy for Sm in the Sm-Co-Fe ternary system on the phase stability and phase relations is interesting, particularly in the Sm-impoverished portion.

The Sm-Co binary system contains SmCo_2 , SmCo_3 , Sm_2Co_7 , $\text{Sm}_5\text{Co}_{19}$, SmCo_5 and $\text{Sm}_2\text{Co}_{17}$ phases with $\text{Sm} \leq 33.3\%$ (mole fraction)^[7, 8]. However, there is no $\text{Dy}_5\text{Co}_{19}$ phase in the Dy-Co system^[9]. The Sm-Fe system possesses SmFe_2 , SmFe_3 and $\text{Sm}_2\text{Fe}_{17}$

phases. Four phases, DyFe_2 , DyFe_3 , $\text{Dy}_6\text{Fe}_{23}$, $\text{Dy}_2\text{Fe}_{17}$, exist in the Dy-Fe system^[10]. Schneider et al^[11] and Hening et al^[12] reported the phase diagram of the Sm-Fe-Co ternary system and found five intermetallic phases, $\text{Sm}(\text{Fe}, \text{Co})_2$, $\text{Sm}(\text{Fe}, \text{Co})_3$, $\text{Sm}_2(\text{Fe}, \text{Co})_7$, $\text{Sm}(\text{Fe}, \text{Co})_5$ and $\text{Sm}_2(\text{Fe}, \text{Co})_{17}$ in the isothermal section at 1 073 K. The complete miscibility of Fe and Co for $\text{Sm}(\text{Fe}, \text{Co})_2$, $\text{Sm}(\text{Fe}, \text{Co})_3$, $\text{Sm}_2(\text{Fe}, \text{Co})_{17}$ phases is confirmed, but no $\text{Sm}_5(\text{Fe}, \text{Co})_{19}$ phase exists in the system^[11, 12]. The phase diagram of the Dy-Co-Fe ternary system has not been published. In this paper, the phase diagram of R-Fe-Co pseudoternary system with $R \leq 33.3\%$ (mole fraction, $R = \text{Sm}_{0.5}\text{Dy}_{0.5}$) is investigated.

2 EXPERIMENTAL

The purity of the raw material was 99.9% for Sm, Dy, Co and 99.8% for Fe. Alloy specimens were prepared in a magnet-controlled arc furnace under high purity argon. To compensate for evaporation losses of Sm during melting, 10% more of Sm was added in excess of the stoichiometric amount. The mass loss of each sample was controlled below 1%. 45 alloy buttons were prepared. The as-cast samples wrapped in Mo foil were sealed in silica tube filled with high purity argon. Specimens with $R \geq 25\%$ (mole fraction) were homogenized at 1 073 K for 20 d and then quenched in water. Specimens with $R \leq 22.2\%$ (mole fraction) were first homogenized at 1 273 K for 5 d and then at 1 073 K for 15 d. After

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homogenizing, they were quenched in water.

Characterization of the specimens was performed using optical microscopy. The etchant used was 2% nital. Differential thermal analysis was done in an LCP-1-type high temperature differential thermal dilatometer under purified argon using alumina crucibles. X-ray diffraction analysis was carried out in a D/max-rA diffractometer equipped with a pyrolytic graphite monochromator. $\text{CuK}\alpha$ radiation was used and the specimens were ground to powders under ethanol. Electron probe microanalysis (EPMA) was performed in a Camebax-micro analyser.

3 RESULTS AND DISCUSSION

3.1 Intermetallic phases

Based on the results of metallographic examination, X-ray diffraction analysis and EPMA, seven intermetallic phases were found: $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_2$, $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_3$, $(\text{Sm}, \text{Dy})_6(\text{Fe}, \text{Co})_{23}$, $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_7$, $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_5$, $\text{Th}_2\text{Ni}_{17}$ -type and $\text{Th}_2\text{Zn}_{17}$ -type $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_{17}$.

1) $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_2$

According to Sm-Fe and Dy-Fe systems, SmFe_2 and DyFe_2 phases are stable. X-ray diffraction analysis confirms that the $(\text{Sm}, \text{Dy})\text{Fe}_2$ phase with MgCu_2 -type cubic structure exists in RFe_2 alloy. However, metallographic examination found that there is a small amount of second phase, which is confirmed as $(\text{Sm}, \text{Dy})\text{Fe}_3$ by EPMA. This result implies that the homogeneity range of $(\text{Sm}, \text{Dy})\text{Fe}_2$ phase is probably shifted slightly to the rare earth-rich side of the ideal stoichiometric ratio. Mei et al.^[13] investigated the $(\text{Tb}_{0.3}\text{Dy}_{0.7})\text{-Fe}$ vertical section and found that the $(\text{Tb}, \text{Dy})\text{Fe}_2$ phase region obviously deviates from the stoichiometric Laves phase composition. Similar situation has been found in $(\text{Dy}, \text{Tb}, \text{Pr})\text{Fe}_2$ compound^[14]. When Co is substituted for Fe, the second phase $(\text{Sm}, \text{Dy})\text{Fe}_3$ clearly decreases and $\text{R}(\text{Fe}_{1-x}\text{Co}_x)_2$ alloys are nearly a single $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_2$ phase in the range of $0.1 \leq x \leq 1$.

The composition dependence of the lattice parameter of the $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_2$ phase for $\text{R}(\text{Fe}_{1-x}\text{Co}_x)_2$ alloys is shown in Fig. 1. It decreases with increasing Co content in the range of $0 \leq x \leq 1$. The decrease in the lattice parameter is attributed to the small size of Co atom. This result is similar to that previously reported in Ref. [3] or Ref. [4].

2) $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_3$

From Sm-Fe, Sm-Co, Dy-Fe and Dy-Co systems, SmFe_3 , SmCo_3 , DyFe_3 and DyCo_3 phases are stable. X-ray diffraction analysis confirms that the $(\text{Sm}, \text{Dy})\text{-Fe}_3$ phase exists in RFe_3 alloy. When Co is substituted for Fe, $\text{R}(\text{Fe}_{1-x}\text{Co}_x)_3$ alloys are a single $(\text{Sm}, \text{Dy})\text{-(Fe, Co)}_3$ phase with PuNi_3 -type structure up to $x = 1$.

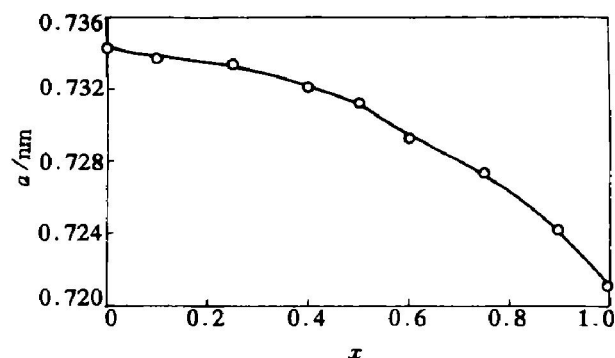


Fig. 1 Composition dependence of lattice parameter of $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_2$ phase in $\text{R}(\text{Fe}_{1-x}\text{Co}_x)_2$ alloys

3) $(\text{Sm}, \text{Dy})_6(\text{Fe}, \text{Co})_{23}$

$\text{Dy}_6\text{Fe}_{23}$ phase is stable only in the Dy-Fe binary system and is of the $\text{Th}_6\text{Mn}_{23}$ -type cubic structure. X-ray diffraction analysis found the $(\text{Sm}, \text{Dy})_6\text{Fe}_{23}$ phase in R_6Fe_{23} alloy is stable (Fig. 2). When Co is substituted for Fe, $\text{R}_6(\text{Fe}_{1-x}\text{Co}_x)_{23}$ alloys are a single $(\text{Sm}, \text{Dy})_6(\text{Fe}, \text{Co})_{23}$ phase up to $x = 0.45$. The $\text{R}_6(\text{Fe}_{0.5}\text{Co}_{0.5})_{23}$ alloy consists of $(\text{Sm}, \text{Dy})(\text{Fe}, \text{Co})_3$ and $(\text{Sm}, \text{Dy})_6(\text{Fe}, \text{Co})_{23}$ phases as well as a small amount of $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_{17}$ phase. It seems that the $(\text{Sm}, \text{Dy})_6(\text{Fe}, \text{Co})_{23}$ phase dissolves 36% Co (mole fraction) at 1073 K.

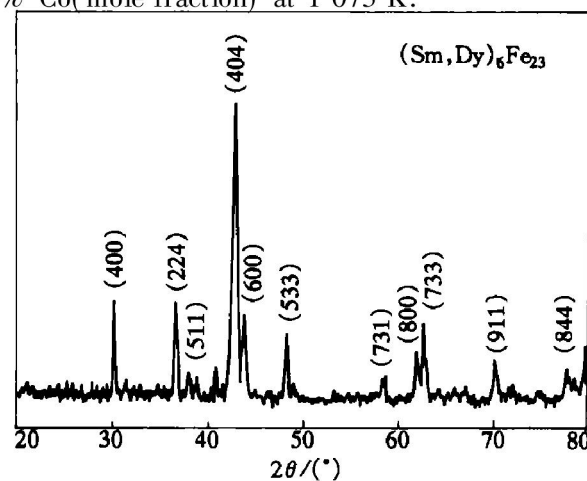


Fig. 2 X-ray diffraction pattern of $(\text{Sm}, \text{Dy})_6\text{Fe}_{23}$ in R_6Fe_{23} alloy

4) $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_7$

The Sm_2Co_7 and Dy_2Co_7 phases are stable in the Sm-Co and Dy-Co systems, respectively, and are of a hexagonal Ce_2Ni_7 structure. When Dy is substituted for Sm, the $(\text{Sm}, \text{Dy})_2\text{Co}_7$ phase in R_2Co_7 alloy is stable in the R-Co system. When Fe is substituted for Co, the microstructure of $\text{R}_2(\text{Fe}_{1-x}\text{Co}_x)_7$ alloys is nearly a single $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_7$ phase in the range of $0.75 \leq x \leq 1$ (Fig. 3). The X-ray diffraction analysis and EPMA confirm that $\text{R}_2(\text{Fe}_{0.3}\text{Co}_{0.7})_7$ alloy consists of $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_7$ and $(\text{Sm}, \text{Dy})_2(\text{Fe}, \text{Co})_{17}$ as well as small amount of $(\text{Sm}, \text{Dy})(\text{Fe},$

Co)₃ phases. Ref. [11] reported that the Sm₂(Fe, Co)₇ phase in Sm-Fe-Co alloys dissolves less than 4% Fe (mole fraction) at 1 473 K and Fe is not found in the Sm₂(Fe, Co)₇ phase in the 95Co5Fe/Sm diffusion couple annealed at 1 073 K. It means that the substitution of Dy for Sm is beneficial to the formation of (Sm, Dy)₂(Fe, Co)₇ phase in R₂(Fe_{1-x}Co_x)₇ alloys. Therefore, the (Sm, Dy)₂(Fe, Co)₇ phase in R₂(Fe_{1-x}Co_x)₇ alloys dissolves about 19% Fe (mole fraction) at 1 073 K.

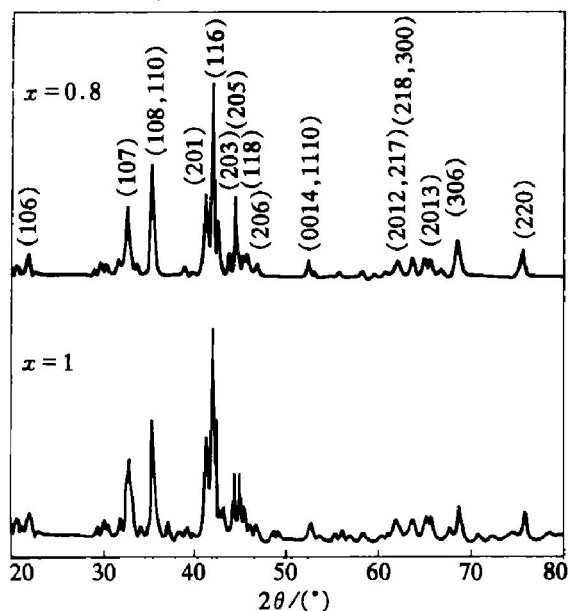


Fig. 3 X-ray diffraction patterns of (Sm, Dy)₂(Fe, Co)₇ in R₂(Fe_{1-x}Co_x)₇ alloys

5) (Sm, Dy)(Fe, Co)₅

The (Sm, Dy)Co₅ phase in RCo₅ alloy is stable and is of a hexagonal CaCu₅-type structure. When Fe is substituted for Co, the microstructure of R(Fe_{1-x}Co_x)₅ alloys is nearly a single phase in the range of 0.9 ≤ x ≤ 1. However, the microstructure of R(Fe_{0.2}Co_{0.8})₅ alloy consists of (Sm, Dy)(Fe, Co)₅, (Sm, Dy)₂(Fe, Co)₁₇ and a small amount of (Sm, Dy)₂(Fe, Co)₇ phases. It means that the Fe solubility in (Sm, Dy)(Fe, Co)₅ phase is nearly 8% at 1 073 K, which is similar to that reported in Ref. [11].

6) (Sm, Dy)₂(Fe, Co)₁₇

From the Sm-Fe, Sm-Co, Dy-Fe and Dy-Co systems, there are Sm₂Fe₁₇, Sm₂Co₁₇, Dy₂Fe₁₇ and Dy₂Co₁₇ phases. The (Sm, Dy)₂Fe₁₇ phase in R₂Fe₁₇ alloy is stable and is of an hexagonal Th₂Ni₁₇-type structure. This result is different from that of Sm₂Fe₁₇ or (Sm, Pr)₂Fe₁₇^[15], which is of rhombohedral Th₂Zn₁₇-type structure. It seems that the substitution of Dy for Sm is beneficial to forming the phase with the Th₂Ni₁₇-type structure. When Co is substituted for Fe, the (Sm, Dy)₂(Fe, Co)₁₇ phase with the Th₂Ni₁₇-type structure in R₂(Fe_{1-x}Co_x)₁₇ alloys is stable when x ≤ 0.2. However, the (Sm,

Dy)₂(Fe, Co)₁₇ phase possesses the Th₂Zn₁₇-type structure when x > 0.3. Both the Th₂Ni₁₇-type and Th₂Zn₁₇-type structures coexist in the range of 0.2 ≤ x < 0.3.

3.2 Isothermal section at 1 073 K

The 1 073 K isothermal section of the R-Fe-Co pseudoternary system with R ≤ 33.3% (mole fraction) is shown in Fig. 4. This isothermal section consists of nine single-phase regions: (Sm, Dy)(Fe, Co)₂, (Sm, Dy)(Fe, Co)₃, (Sm, Dy)₆(Fe, Co)₂₃, (Sm, Dy)₂(Fe, Co)₇, (Sm, Dy)(Fe, Co)₅, Th₂Ni₁₇-type and Th₂Zn₁₇-type (Sm, Dy)₂(Fe, Co)₁₇, αFe and αCo; 14 two-phase regions: (Sm, Dy)(Fe, Co)₂ + (Sm, Dy)(Fe, Co)₃, (Sm, Dy)(Fe, Co)₃ + (Sm, Dy)₆(Fe, Co)₂₃, (Sm, Dy)(Fe, Co)₃ + (Sm, Dy)₂(Fe, Co)₇, (Sm, Dy)₂(Fe, Co)₇ + (Sm, Dy)(Fe, Co)₅, (Sm, Dy)(Fe, Co)₃ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇, (Sm, Dy)(Fe, Co)₅ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇, (Sm, Dy)₆(Fe, Co)₂₃ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇, (Sm, Dy)₂(Fe, Co)₇ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇, Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + Th₂Zn₁₇-type (Sm, Dy)₂(Fe, Co)₁₇, Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + αFe, Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + αFe, Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + αCo and αFe + αCo; and six three-phase regions: (Sm, Dy)(Fe, Co)₃ + (Sm, Dy)₆(Fe, Co)₂₃ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇, (Sm, Dy)(Fe, Co)₃ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + (Sm, Dy)₂(Fe, Co)₇, (Sm, Dy)₂(Fe, Co)₇ + (Sm, Dy)(Fe, Co)₅ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇, (Sm, Dy)₆(Fe, Co)₂₃ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + Th₂Zn₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + αFe and Th₂Ni₁₇-type (Sm, Dy)₂(Fe, Co)₁₇ + αFe + αCo. Compared with the Sm-Fe-Co ternary system reported in Ref. [11], the isothermal section of R-Fe-Co pseudoternary system with R ≤ 33.3% (mole fraction) contains (Sm, Dy)₆(Fe, Co)₂₃ phase, and the homogeneity range of (Sm, Dy)₂(Fe, Co)₇ phase for the R-Fe-Co system is larger than that of Sm₂(Fe, Co)₇ phase for the Sm-Fe-Co system.

3.3 Vertical section of RFe₂-RCo₂

Based on the results of DTA, OM and XRD, a vertical section of RFe₂-RCo₂ in R-Fe-Co pseudoternary system is tentatively drawn in Fig. 5. It consists of two single-phase regions: L and (Sm, Dy)(Fe, Co)₂; two two-phase regions: L + (Sm, Dy)(Fe, Co)₃ and L + (Sm, Dy)₂(Fe, Co)₇ and two three-

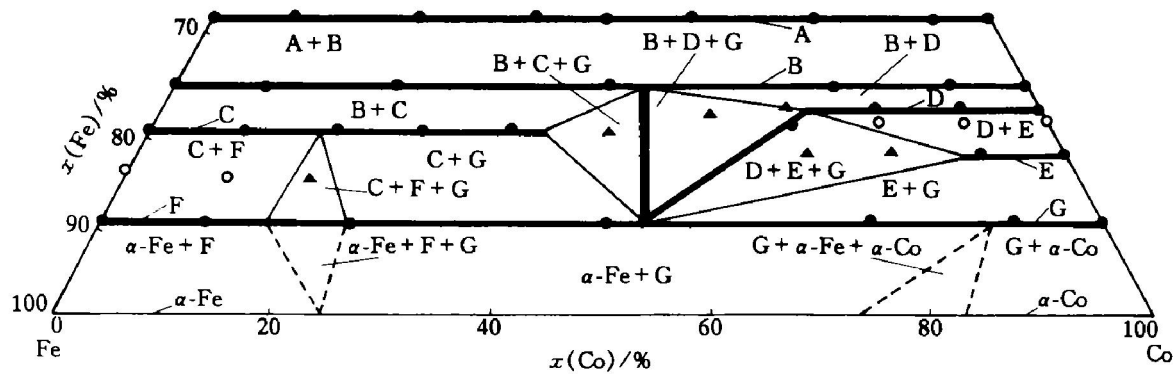


Fig. 4 Isothermal section of R-Fe-Co pseudoternary system with

$R \leq 33.3\%$ (mole fraction) at 1073 K

A = (Sm, Dy)(Fe, Co)₂; B = (Sm, Dy)(Fe, Co)₃; C = (Sm, Dy)₆(Fe, Co)₂₃; D = (Sm, Dy)₂(Fe, Co)₇;
E = (Sm, Dy)(Fe, Co)₅; F = Th₂Ni₁₇ type (Sm, Dy)₂(Fe, Co)₁₇; G = Th₂Zn₁₇ type (Sm, Dy)₂(Fe, Co)₁₇
●—Single phase region; ○—Two phase region; ▲—Three phase region

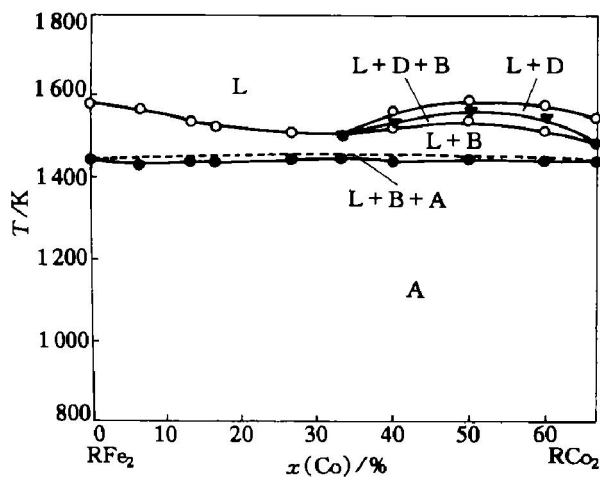


Fig. 5 Vertical section of RFe₂-RCO₂ in R-Fe-Co pseudoternary system

A = (Sm, Dy)(Fe, Co)₂; B = (Sm, Dy)(Fe, Co)₃;
C = (Sm, Dy)₂(Fe, Co)₇

phase regions: L + (Sm, Dy)(Fe, Co)₃ + (Sm, Dy)₂(Fe, Co)₇ and L + (Sm, Dy)(Fe, Co)₂ + (Sm, Dy)(Fe, Co)₃.

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