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Recent developments in RFe₁₂-type compounds for permanent magnetsA.M. Gabay^{*}, G.C. Hadjipanayis

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ABSTRACT

This article discusses the recently intensified studies on the ThMn₁₂-type hard magnetic compounds. Among the important developments were the synthesis of compounds superior to the Nd₂Fe₁₄B in saturation magnetization, the increased interest on Ce-substituted and rare-earth-free compounds and also the emergence of mechano-chemistry as a method for manufacturing anisotropic powders with high coercivity. The theoretical studies might have had a greater impact if more attention was paid to the structural stability of these compounds. Little information is available on the fabrication of dense three-dimensional magnets; these efforts must be intensified for a successful implementation of the most advanced RFe₁₂-type materials.

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1. Introduction

The tetragonal crystal structure named after its ThMn₁₂ prototype features one position for a larger atom (usually the rare earth) and three positions for the smaller atoms (e.g., Fe and a third element which stabilizes the structure); it is schematically shown in Fig. 1. The 8i sites are strongly preferred by the structure-stabilizing elements which are the large transition metal atoms (Ti, V, Nb, Mo, Ta, W); Al and Si, which also stabilize the structure, prefer the 8f sites [1,2]. A light element atom (H, N, C) may occupy the interstitial positions, although the resulting compound can only exist as metastable.

The potential of the ThMn₁₂-type compounds as permanent magnet materials was recognized in the late 1980s with the discovery of such compounds containing 75 and more at.% Fe [1]. From the number of scientific publications referring to this structure (Fig. 2), the current interest on these materials may appear modest compared to its peak in the 1990s. However, the last four years saw a dramatic increase in references to the ThMn₁₂-type compounds in the context of permanent magnet development. One reason for this renewed, application-focused interest is that after three decades of intensive development, the maximum energy product (BH)_{max} of the best available magnets, Nd-Fe-B, is finally approaching its theoretical limit of 520 kJ m⁻³. Thus, superior materials are now particularly welcome. The second reason is a growing demand for energy-dense magnets operating at 175–200 °C, the demand driven by proliferation of the wind turbines and electric (hybrid) vehicles. Both the Nd-Dy-Fe-B and Sm-Co magnets operating at these temperatures have significant, albeit different, shortcomings. The third

reason – and the actual trigger for the renewed interest – was the 2010–11 rare earth supply crisis which demonstrated how risky the current reliance on just a few rare earth elements (Nd, Pr and especially Dy) may be.

Some researchers see in the R(Fe,M)₁₂ compounds and their interstitially modified derivatives possible solutions to the above challenges. Provided a minimal amount of the structure-stabilizing M element is used, these compounds may offer us even greater (BH)_{max} than the Nd₂Fe₁₄B compound. When moderately alloyed with Co or/and modified interstitially with the light elements, these compounds also demonstrate higher Curie temperatures and, presumably, better temperature stability. Compared to the typical Nd-Fe-B and Sm-Co magnets, the R(Fe,M)₁₂ compounds are rare-earth-lean, some are actually rare-earth-free. It is also worth noting that thanks to their versatility the number of unexplored R(Fe,M)₁₂ compositions – including those with multiple R and M elements – is still very large. In this Viewpoint article, we review and analyze the reports about these compounds generated in the last four years in attempt to identify the most promising developments and to suggest what we see as desirable changes to specific approaches.

2. Ab initio studies and computational screening

In line with the general trend in new materials search, first-principle calculations based on the density functional theory are now addressing the specific aspects of the magnetism of the RFe₁₂-type compounds with a sufficient accuracy providing guidance to experimentalists. The theoretical finding by Miyake et al. [3] that the Ti atom decreases the saturation magnetization of the NdFe₁₁Ti compound more than could be expected for simple dilution of the magnetic Fe atoms was cited by

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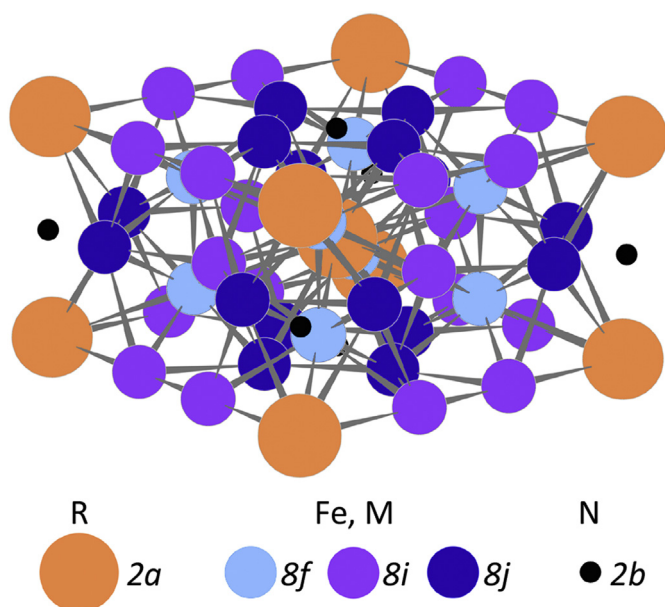


Fig. 1. Tetragonal unit cell of $R(Fe,M)_{12}$ lattice containing two formula units. Also shown are the nitrogen positions in interstitially modified $R(Fe,M)_{12}N$.

Hirayama et al. [4] as a motivation for the synthesis of a binary $NdFe_{12}$ compound (see next section). Although ab initio modelling of the finite-temperature magnetism remains challenging, the approach proposed recently by Matsumoto et al. [5] was successful enough to single out enhancement of the 4f-3d indirect exchange coupling as a way to improve the high-temperature properties of the RFe_{12} -type compounds.

Several theoretical works compared the effects of different light elements introduced interstitially. That included the effects of elements like O and F [6,7] which would be very difficult to study experimentally. For the compounds containing inexpensive cerium, Ke and Johnson [8] predicted improved hard magnetic properties in carbon-modified $Ce(Fe_{1-x}Co_x)_{11}TiC$ compounds when $0.7 < x < 0.9$.

In a more ambitious attempt of computational screening reported by Körner et al. [9], as many as 1280 compounds were generated and analyzed automatically. In the future, such theoretical high-throughput

screening is likely to become an important tool for materials development; however, the work by Körner et al., in our opinion, highlights the specific risks associated with this approach. The inaccurate assumption about the site preferences (the atoms substituting for Fe were allowed to occupy only the 8i site; this deficiency was partially addressed in a follow-up work [10]) and the rather narrow approach (exactly one Fe atom per formula unit could be substituted, no Zr atoms were allowed in the 2a sites, etc.) mean that promising materials could be left undetected. Moreover, many “false positives” are inevitably generated if conclusions about the stability of compounds are based only on their respective formation energies. Instead, the formation energy of the compound should be compared to that of all possible alternatives. In the case of the $R(Fe,M)_{12}$ where R is the rare earth, the most likely alternative is a mixture of bcc Fe-M and $R_2(Fe,M)_{17}$ phases. Harashima et al. [11] claimed that the $NdFe_{11}M$ is stable when M is represented by magnetic Co (thus exhibiting a very high magnetization) based on comparison with a mixture of the Fe-Co and Nd_2Fe_{17} phases. The correct comparison would have been with a mixture of the Fe-Co and $Nd_2(Fe,Co)_{17}$; phase equilibria studies [12] so far have failed to detect a stable $NdFe_{11}Co$ compound. Similarly doubtful is the theoretical prediction of a stable $CeFe_{10}Si_2$ compound by Drebov et al. [13]; the compound was detected in melt-spun alloys [14], but not in arc-melted samples [15].

3. RFe_{12} and $R(Fe,Co)_{12}$ compounds with high magnetization

Although binary RFe_{12} compounds do not exist in the bulk form, it has been long known that $SmFe_{12}$ could be prepared as a *thin film* [16–20]. Recently, Hirayama et al. [4,21] and Sato et al. [22] obtained highly textured films of the compounds $NdFe_{12}$ (subsequently converted into $NdFe_{12}N_x$), $SmFe_{12}$ and $Sm(Fe,Co)_{12}$ by growing them epitaxially on W- or V-buffered single-crystal MgO substrates. The experimentally determined magnetic properties for these compounds are presented in Table 1; the saturation magnetizations $\mu_0 M_s$ were found to be higher than in the $Nd_2Fe_{14}B$. The $SmFe_{9.6}Co_{2.4}$ compound is the most impressive candidate for a permanent magnet material: its $(BH)_{max}$ has an upper limit of 630 kJ m^{-3} , whereas both the Curie temperature and anisotropy field are significantly higher than for the $Nd_2Fe_{14}B$. Unfortunately, these thin film materials do not have high coercivity and furthermore thin films cannot serve as permanent magnets. It is also worth noting that even though the epitaxy may be realized in the bulk (e.g., by first preparing core-shell nanoparticles [24]), the volume occupied by the substrate cannot be excluded when determining the energy density of a three-dimensional magnet. Thus, $(BH)_{max}$ of the actual magnet will still be lower than that of the compound.

Unstable compounds like RFe_{12} can sometimes be obtained through non-equilibrium crystallization. Suzuki [25] reported observation of the YFe_{12} structure in multi-phase Fe-Y alloys which had been subjected to melt-spinning. It is conceivable that such approach will allow for synthesis of a reasonably pure RFe_{12} , but only as a metastable polycrystalline material lacking crystallographic texture. Based on past experience, conversion of such material into highly textured magnets fully utilizing the M_s of the compound will be extremely difficult.

Table 1

Curie temperature and room-temperature values of saturation magnetization and anisotropy field for bulk $Nd_2Fe_{14}B$ and RFe_{12} -based epitaxial films.

Compound	T_C (°C)	$\mu_0 M_s$ (T)	$\mu_0 H_a$ (T)	Ref.
$Nd_2Fe_{14}B$	313	1.60	7	[23]
$NdFe_{12}N_x$	≈ 550	1.70	6–8	[4,22]
$SmFe_{12}$	282	1.64	12	[21]
$SmFe_{9.6}Co_{2.4}$	586	1.78	12	[21]

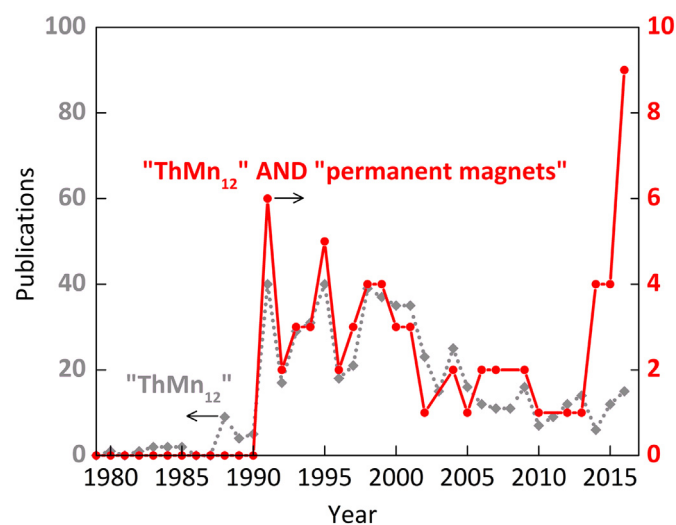


Fig. 2. Annual number of publication records in the Web of Science database on the topic of $ThMn_{12}$ and on the same topic in the context of permanent magnets for the years 1979 through 2016.

4. High-magnetization R(Fe,Co,M)₁₂ compounds through multiple substitutions

According to Kobayashi et al. [26], stabilization of R(Fe,Ti)₁₂ structures when the Ti atoms replace part of the smaller Fe in the 8i sites can be understood as a decrease of local mismatches in interatomic distances unfavorable for the orbital hybridization. A similar effect is believed to occur when part of the rare earth in the 2a sites is replaced with the smaller Zr atoms. A combined alloying with Zr and Ti, thus makes it possible to achieve stability of the structure with less than one Ti atom per formula unit. When combined with replacing 25% of the Fe atoms with Co atoms, this approach was reported [27–29] to allow for fabrication of the Sm_{0.8}Zr_{0.2}(Fe,Co)_{11.5}Ti_{0.5} and Nd_{0.7}Zr_{0.3}(Fe,Co)_{11.5}Ti_{0.5}N_x compounds exhibiting M_s nearly equal or even exceeding that of Nd₂Fe₁₄B (Table 2). However, these compounds were observed alongside with other phases including, in all reported cases, the ferromagnetic α -(Fe,Co) phase. Furthermore, the alloys, which had been prepared via strip-casting, were characterized with their crystallites being oriented randomly. Thus, there are certain doubts regarding the minimum sufficient amount of Ti and the estimated values of the anisotropy field. We attempted to replicate the synthesis of the Sm_{1-x}Zr_x(Fe,Co)_{12-y}Ti_y alloys using arc-melting followed by a homogenization, but we could not obtain a single phase for $y < 0.65$. Characterization of the single-phase alloy with $y = 0.65$ (Fig. 3) revealed, in addition to a T_C of 570 °C, strong anisotropy and $\mu_0 M_s$ of 1.47 T (Table 2). These properties are markedly higher than in the case of the SmFe₁₁Ti, thus confirming the general validity of the approach, even if the $\mu_0 M_s$ attainable in the *single phase* is not as high as reported by Kuno et al. [27]. Stabilization of the RFe₁₂-based compounds through simultaneous substitutions for both R and Fe should certainly be addressed in future ab initio studies – and included in future computational screenings.

5. RFe₁₂-type compounds with less-critical rare earths or with no rare earths

Cerium, despite being considered as critical element [32], is by far the most abundant and least expensive of the rare earths. It is therefore attractive for development of moderately strong (160–280 kJ m⁻³) permanent magnets which would be operational up to a temperature around 150 °C; most magnets of that grade currently rely on the highly demanded Nd. Because of the mixed valent state of Ce atoms, most of the Ce(Fe,M)₁₂ compounds exhibit low values of T_C and M_s ; the T_C of the CeFe_{10.5}Mo_{1.5}, for instance, is only 68 °C [33]. The CeFe₁₀Si₂ is an exception exhibiting a T_C as high as 310 °C [14]. Its anisotropy, however, is rather weak ($\mu_0 H_a = 2.2$ T), and there are no indications that a significant coercivity could be developed through melt-spinning [34]. The relatively large atomic radius of Ce indicates that the CeFe₁₀Si₂ is likely to be unstable [35], but it can be stabilized through Zr substitution for Ce [15].

The low Curie temperature may also be increased by replacing part of Fe with Co. A high-throughput screening based on synthesis of heterogeneous non-equilibrium states with analysis by microscopic methods [36] identified CeFe₉Co₂Ti as promising compound; however, a more direct characterization [37,38] found that the corresponding anisotropy field was overestimated. According to Zhou et al. [37], a CeFe₁₀CoTi exhibits fairly high values of T_C (312 °C) and $\mu_0 M_s$ (1.21 T).

Table 2

Room-temperature saturation magnetization and anisotropy field reported for Sm(Fe,Ti)₁₂ and Nd(Fe,Ti)₁₂N_x compounds modified with Co and Zr.

Compound	$\mu_0 M_s$ (T)	$\mu_0 H_a$ (T)	Ref.
SmFe ₁₁ Ti	1.14–1.22	8.3–10.5	[30,31]
Sm _{0.92} Zr _{0.08} (Fe _{0.75} Co _{0.25}) _{11.35} Ti _{0.65}	1.47	>9.0	This work
Sm _{0.8} Zr _{0.2} (Fe _{0.75} Co _{0.25}) _{11.5} Ti _{0.5}	1.58	7.4	[27]
Nd _{0.7} Zr _{0.3} (Fe _{0.75} Co _{0.25}) _{11.5} Ti _{0.5} N _x	1.67	6.6	[28,29]

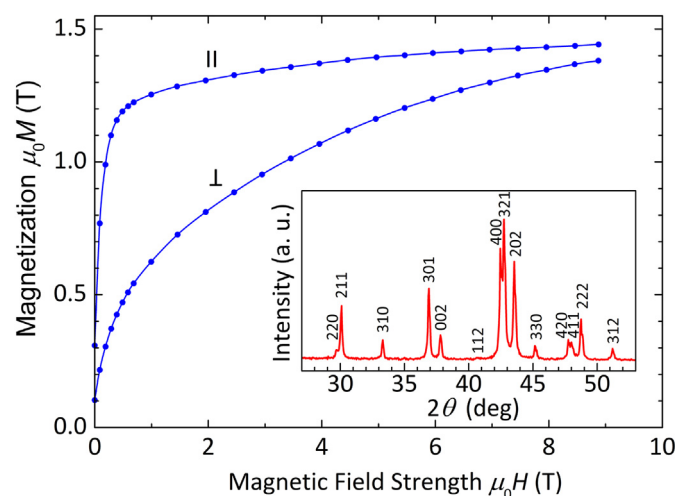


Fig. 3. Room-temperature magnetization curves of oriented Sm_{0.92}Zr_{0.08}(Fe_{0.75}Co_{0.25})_{11.35}Ti_{0.65} powder. Inset shows X-ray diffraction spectrum of the randomly oriented powder with the CuK α radiation. The alloy was prepared by arc-melting and subsequently homogenized at 1100 °C for 25 h.

Still, the low $\mu_0 H_a$ of only 1.7 T did not allow for a high coercivity in the nanocrystalline alloys prepared via melt-spinning, with a $(BH)_{\max}$ not exceeding 12 kJ m⁻³ [39]. The only known remedy for the weak anisotropy the Ce(Fe,M)₁₂ compounds is to replace part of Ce with Sm [38, 40,41]. Despite being scarcer than Ce, Sm does not cost as much as Nd; according to Nassar et al. [42], Sm is less vulnerable to supply restriction than any other rare earth metal.

Zirconium substitution for the rare earth was already mentioned in the context of stabilization of the R(Fe,Co,Ti)₁₂ structures; the large Zr atoms occupy exclusively the “rare-earth” 2a sites [43,44]. Although this element is more expensive than Ce or Sm, it is not considered critical. The largest amount of Zr can be accommodated in the R_{1-x}Zr_x(Fe,M)₁₂ compounds with M = Si [35]. In 2016, our group reported the synthesis of a ZrFe₁₀Si₂ [15] – the first rare-earth- and actinide-free ThMn₁₂-type compound exhibiting a room-temperature ferromagnetism. This *metastable* compound exhibits a moderately high $\mu_0 M_s$ up to 1.15 T (for the ZrFe_{10.4}Si_{1.6} composition; see Fig. 4). Unfortunately, its uniaxial magnetocrystalline anisotropy is rather weak, $\mu_0 H_a \leq 1.9$ T, so the maximum coercivity developed in melt-spun alloys was only 0.7 kOe [45]. The latest experiments indicated that the quaternary compounds ZrFe₁₀M_{0.4}Si_{1.6} are *stable* at 1100 °C when M = V, Cr, Mo and W; their

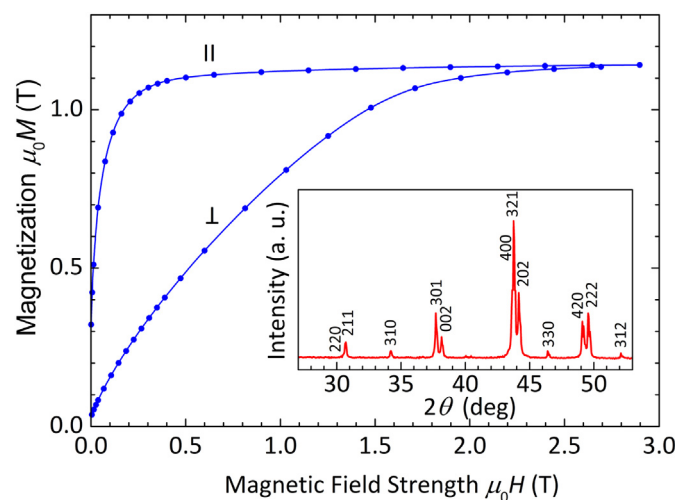


Fig. 4. Room-temperature magnetization curves of oriented Zr_{1.1}Fe_{10.4}Si_{1.6} powder. Inset shows X-ray diffraction spectrum of the randomly oriented powder with the CuK α radiation. The alloy was prepared by arc-melting.

anisotropy, however, is still weak. Of certain interest may be the very rare-earth-lean $R_{1-x}Zr_x(Fe, Si)_{12}$ compounds with $R = Nd$ [46] and Sm [45]. The $Sm_{0.3}Zr_{0.7}Fe_{10}Si_2$ compound containing as little as 6.2 wt% Sm is a viable permanent magnet material characterized by a magnetic hardness parameter [47] of 1.31 and a theoretical $(BH)_{max}$ up to 278 kJ m^{-3} .

6. Manufacturing of magnets

Most of the reports about obtaining substantial coercivity H_c in the RFe_{12} -type materials involve nanocrystalline alloys which are particularly suitable for this task [48–53]. Still, the reported H_c values are typically <10% of the corresponding anisotropy field; clearly, practical realization of the hard magnetic potential of the $R(Fe, M)_{12}$ compounds is much more difficult than it was for the $Sm-Co$ and $Nd-Fe-B$. The phases surrounding the typical $R(Fe, M)_{12}$ compounds in the equilibrium phase diagram are ferromagnetic and thus cannot provide magnetic insulation for the $R(Fe, M)_{12}$ crystallites. Also they usually melt at relatively high temperatures making it difficult to fabricate anisotropic magnets through liquid-phase sintering. Unfortunately, very little information is available on any research efforts addressing this critically important subject. It is quite possible that such efforts are indeed under way with any successful results being withheld as proprietary and with failures being though unworthy of sharing. A patent application [54] filed by Toyota in 2016 includes examples describing how anisotropic $Sm_{1-x}Zr_x(Fe, Co, Ti)_{12}$ magnets exhibiting good properties *had been prepared* using $Sm_5(Fe, Ti)_{17}$ and $Sm(Co, Cu)_5$ alloys as the sintering aid. This is certainly an intriguing possibility, although we are not ready to accept these examples as a sound report.

The remanent magnetization of nanocrystalline alloys prepared via melt-spinning or intensive milling is lowered due to the lack of grain texture. Reduction-diffusion of mechanically activated oxides, also known as mechanochemical synthesis, allows for similar coercivity in single crystal particles which can be oriented – a necessary condition for realizing the theoretical $(BH)_{max}$. Gabay et al. [38,55] prepared anisotropic powders of several $R(Fe, Co)_{11}Ti$ and $R_{1-x}Zr_xFe_{10}Si_2$ compounds by reducing with Ca mechanically activated mixtures of rare earth oxides, ZrO_2 , Fe_2O_3 , TiO_2 and Si . An oriented $Sm_{0.7}Zr_{0.3}Fe_{10}Si_2$ powder exhibited μ_0H_c of 1.08 T and full-density-projected $(BH)_{max}$ of 110 kJ m^{-3} . Also, a Sm -lean powder with composition $Sm_{0.3}Ce_{0.3}Zr_{0.4}Fe_{10}Si_2$ was prepared with μ_0H_c of 0.52 T and $(BH)_{max}$ of 81 kJ m^{-3} . A reduction-diffusion of *chemically synthesized* precursors may have an even greater potential, because this method is more suitable for a large-scale production. Attempted in the past for $NdFe_{10}Mo_2N_x$ [56], this approach was more recently developed for manufacturing high-quality $Sm_2Fe_{17}N_x$ powders [57]. Currently, both the $Sm_2Fe_{17}N_x$ and $R(Fe, Mo)_{12}N_x$ powders are intended for polymer-bonded magnets [58]; the former demonstrate superior magnetic properties, whereas the latter, in addition to the lower rare-earth content exhibit a better corrosion resistance.

The $R(Fe, M)_{12}N_x$ nitrides are particularly difficult to convert into reasonably dense magnets. Because the Gibbs energy of these compounds is higher than that of the mixture of the respective RN nitrides and bcc Fe phase, diffusion of atoms larger than the N atoms enables not only densification, but also the formation of stable RN nitrides, leading to decomposition of the hard magnetic compound. A compromise may still be possible: after detailed investigation of the thermodynamics and kinetics of the decomposition Kong et al. [59] were able to prepare >90%-dense $Ce_{0.2}Nd_{0.8}Fe_{10.5}Mo_{1.5}N_{1.3}$ magnets by subjecting the nanocrystalline powders to spark-plasma sintering at 600–650 °C. Also promising may be the *dynamic* compression which was successfully used to compact the metastable $Sm_2Fe_{17}N_x$ powders [60].

7. Conclusions

Both the metastable nitrides and some thermodynamically stable, particularly Co -substituted, RFe_{12} -type compounds possess intrinsic magnetic properties superior to those of the $Nd_2Fe_{14}B$ in the

temperature range of 150–200 °C. Stabilization of the $RFe_{12-y}M_y$ compounds with a minimal substitution for Fe requires more focused theoretical studies. In order to be really helpful, the computational screening efforts should evaluate the structural stability of compounds beyond the values of their respective formation energies. Limited Ce and Zr substitutions may help to stabilize the $R(Fe, M)_{12}$ structure or to decrease its reliance on certain critical elements; it is very unlikely, that compounds with R represented by only Ce and/or Zr will find application in the permanent magnets. Mechanochemistry and, possibly, chemistry may be considered for manufacturing high quality powders for anisotropic bonded magnets. More efforts need to be dedicated to metallurgical aspects of densification of the RFe_{12} -type powders; these efforts will be critical for the successful development of high-energy magnets.

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