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Graphical Abstract

ABSTRACT

In this study, fabrication of Pr$_{15-x}$Dy$_x$Fe$_{77}$B$_8$ (at.%) alloys with $x = 0, 1, 2,$ and 3 compositions was performed with a mini vacuum arc melting furnace. The alloys were successively annealed in an inert atmosphere for microstructure homogenization. All alloys exhibit the typical characteristics of a permanent magnet. The coercivity gradually increases with increasing atomic fraction of Dy. The heat treatment applied to the samples changed the properties from those of a permanent magnet to those of a microwave absorber. The reflection loss (RL) value evaluated by a Vector Network Analyzer (VNA) shows broadband absorption characteristics in the full 8 GHz–12 GHz frequency range. The RL and bandwidth increase with the addition of the Dy substitute. The as-cast and annealed Pr$_{15-x}$Dy$_x$Fe$_{77}$B$_8$ alloy with the $x = 1$ composition shows superior absorption, starting from the 8 GHz frequency and increasing progressively, reaching less than -17 dB or an absorption of more than 85.0 % at a peak frequency of 10.5 GHz, after which it weakens to -6 dB at a frequency close to 12 GHz. Heat-treated samples were characterized by absorption with two almost overlapping absorption peaks at frequencies of 9.5 GHz and 11.5 GHz, leading to very broadband absorption across the full frequency range (8-12 GHz). It is concluded that permanent magnetic materials could be converted into radar absorbing materials (RAM). RAM based on heat-treated rare earth permanent magnet has broadband absorption characteristics.
1. Introduction

Since their discovery in the 1990s, rare-earth-based hard magnets with RE₆Fe₁₄B (RE = rare earth) phases, such as neodymium iron boron (Nd-Fe-B) with a Nd₂Fe₁₄B phase [1, 2], have remained the most powerful permanent magnets up to today. Because their magnetic properties are superior to those of other permanent magnets, such as barium hexaferrite [3, 4], alnico [5], and samarium cobalt [6, 7], Nd-Fe-B magnets have received great attention from various researchers. As might be expected, within a relatively short period, Nd-Fe-B permanent magnets were developed very rapidly and reached the last stage of development, a circular economy activity [8, 9]. As a result of such rapid development, assisted by various processing technologies such as rapid solidification [10], powder metallurgy [11], and HDDR [12], permanent magnets with a maximum energy product or (BH)ₘₐₓ close to the theoretical limit of 512 kJ/m³ were produced. According to a recent report [10], the highest (BH)ₘₐₓ value that has been achieved for rare-earth permanent magnets is 474 kJ/m³ [13]. This value is approximately 92.6% of the highest value that can be obtained for Nd-Fe-B permanent magnets.

It is well understood that hard magnetic materials can be converted into radar absorbing materials (RAM), such as hexaferrite, after partial substitution of Fe³⁺ ions with others, like Mn²⁺ and Ti⁴⁺ ions [14], Zn²⁺ and Sn⁴⁺ ions [15], and many more [16, 17]. The partial substitution of Fe³⁺ ions was intended to reduce the magnetocrystalline anisotropy constant K, and hence, the coercivity of ion-substituted hard ferrites decreases with no excessive reduction in the remanence value. Hard magnetic materials such as barium and/or strontium hexaferrite are considered to have potential for RAM because they have a high saturation magnetization of 0.38 T [3] and high electrical resistance. Considering that the material to become electromagnetic wave (EM) absorbers, magnetic properties with a high saturation magnetization (Mₛ) value, but low coercivity (Hᵣ) or anisotropy field (Hᵥₐ) are necessary. In the case of RE permanent magnets, it is therefore appropriate to reduce the Hᵥₐ of the RE₂Fe₁₄B phase, which may be achieved by partial substitution with other RE metals. Yongqiang Xu et al. [18] replaced Nd in the Nd₂Fe₁₄B magnetic phase with La to (Nd₁₋ₓLaₓ)₂Fe₁₄B (x = 0–0.4). The overall objective was to decrease the use of Nd through partial substitution. The findings of this study of this compound show that, material still showing superiority as a permanent magnet. Moreover, it also had the feature of absorbing the EM at a frequency of 2–16 GHz with a single absorption. This partial substitution effect reduced Hᵥₐ and Mₛ. The decrease in Hᵥₐ as x increased, the absorption peak shifted to a lower frequency of 9.8 GHz at x = 0–6.5 GHz at x = 0.4. Simultaneously the reflection loss (RL) value decreased, but with the bandwidth increased. Results of such studies suggest that a reduction in Mₛ has the effect of reducing the RL. The fall in the value of Hᵥₐ changes the position of the absorption peak. In the case of absorption of EM, rare earth compounds characterized by a single peak absorption in the 2–16 GHz frequency range with high absorption characteristics.


dLi Jun et al. [19] reported the use of RE metal for NdₓFe₆(Bₓ₋ₓ)B₁₁ compounds (x = 3–9). This material was cast as a meltspun ribbon. The diffraction pattern consisted of diffraction peaks (110) and (220) belonging to α-Fe, which is unlikely to be a single phase. Other phases need to be present in the form of amorphous phases. All samples (x = 3–9) had magnetic hysterisis loops with very high Mₛ (~150 emu/g) and very low Hᵥₐ values (86.49 Oe – 122.9 Oe). The absorption of EM from this material also consisted of a single absorption peak in the frequency range 2–18 GHz with a relatively high bandwidth. The composition with x = 9 gave the best absorption, reaching 23.9 dB at an absorption frequency of 10.2 GHz; that is, more than 99% of the incoming waves were absorbed. Thus, alloy with x = 9 is recommended as a candidate alloy for use in RAM applications.

In 2020, Jinbo et al. [20] conducted a review of microwave absorbers based on rare earth transition metal alloys as new high-performance microwave absorbers. Magnetic phases like Y₂Fe₁₇, NdFe₁₄Co₁₀V₂ (0 ≤ x ≤ 0.5), with no exception for phase 2-14-1 like Sm₂Fe₁₄B, all of which have the capability to absorb electromagnetic waves. The absorption was characterized by a single absorption with a wide bandwidth in the frequency range of 2-18 GHz. A Sm₂Fe₁₄B compound with a planar anisotropy may have a value of RL = –42 dB at a frequency of 2.9 GHz. Similarly with the Nd₁₋ₓSmₓFe₁₄B (0 ≤ x ≤ 0.5) compound, the value of RL = –40 dB at a frequency of 10 GHz with a sample thickness of only 1.6mm.

Based on the above review, the use of rare earth elements in RAM has been shown to provide excellent microwave absorbing properties. It was shown that the use of rare-earth metals is not limited to permanent magnets but can also be extended to RAM. Zhou et al. [21] and Adi et al. [22] used lanthanum metal for the manufacture of LaMnO₃ as a RAM. The perovskite La₀.₅Ba₀.₅MnO₃ was prepared by the sol-gel process, and its microwave absorption properties in the frequency range of 2–18 GHz were reported. The microwave absorption peak was 13 dB at a frequency of 6.7 GHz, and the effective absorption bandwidth above 10 dB reached 1.8 GHz. Such a high microwave absorption capability of rare-earth-based compounds must be closely related to high magnetic properties, such as high saturation magnetization and remanence, with a low coercivity combined with microwave property parameters such as magnetic permeability and electric permittivity. In this paper, we report results and an analysis based on our research works on PrₓDy₁₋ₓFe₇B₈ (at.%), with x = 0, 1, 2, 3 compositions. The magnetic properties and microwave characteristics of the alloys before and after heat treatment are evaluated by a permagraph and a vector network analyser (VNA), respectively. In addition to the possibility of absorptions in a wide frequency range, we investigated absorption in the x-band only. In other frequency ranges, our facility does not support the evaluation to be carried out.

2. Materials and methods

Magnetic alloys (Pr₁₅₋ₓDyₓ)Fe₇₋ₓB₈ (at.%) with a composition of x = 0, 1, 2, and 3 were prepared using Pr, Dy, Fe, and B precursors, each with a mass according to the designated composition and a total mass of 15 g.

Figure 1. Photos showing (a) alloy fabrication by MAM-1, (b) as-cast ingots and (c) pieces of ingots in sealed quartz tubing.
The RE metals were supplied by Inner Mongolia rare Earth Ovonic Metal Hydride Co., Ltd. Other precursors such as Fe and B were supplied by Merck. All alloy precursors were arc melted under a mini vacuum arc melting furnace (MAM-1) Edmund Bühler GmbH. Melting was carried out at least 6 times with 5 reversals until a homogeneous melting ingot was obtained. During the melting process, the mass loss was so small that it could be neglected; hence, the ingot must possess the same composition as the designated composition.

Figure 1 shows a series of photographs taken during sample alloy preparation by a vacuum arc melting furnace (Figure 1a), while a photograph of as-cast ingots is shown in Figure 1b, and ingots resulting from the casting after undergoing annealing treatment at a temperature of 900 °C for 24 h for grain homogenization are shown in Figure 1c.

Annealing was carried out under an inert atmosphere in which ingots were placed in a quartz tube after evacuation of air, which was filled with argon gas before being sealed off. Pieces of annealed ingots were then ground in a disk milling apparatus for a short vibration time to prevent excessive oxidation. This treatment stage produced a powder with a pass size of 20 microns. The ingot powder was then put into a cylindrical mould with a diameter of 12 mm and pressed uniaxially with a load of 10 tons. One sample of the alloy of each composition in the form of a pellet was immersed in a liquid adhesive, leading to a bonded magnet sample. The remanence and coercivity of all samples were determined from the respective hysteresis loops. Other pellet samples underwent heat treatment with the profile shown in Figure 2.

The capsule containing alloy samples after homogenization at a temperature of 900 °C, were inserted into the programmable furnace. The heat treatment was programmed as follows: heated to 1050 °C and then maintained for 2 h. The furnace temperature was lowered to 900 °C, then maintained for 2 h. Finally, the furnace temperature was reduced to 550 °C for 1 h and then cooled to room temperature.

The phase structure of the powder samples after milling was determined by an XRD Bruker D8 Advance 3 kW equipped with a LynxEye XE-T detector with CuKα radiation (λ = 1.5406 Å) and a step size of 0.01. The XRD data were further quantitatively analysed by General Structure Analysis System (GSAS) software. Furthermore, the surface morphology of the samples was observed under a JEOL JED 350 scanning electron microscope (SEM) equipped with an EDS element analyser. The magnetic properties and microwave characteristics of all samples were evaluated by a Magnet-Physik Permagraph and a vector network analyser (VNA), respectively.

3. Results and discussion

3.1. Microstructural and magnetic properties of Pr\(_{15-x}\)Dy\(_x\)Fe\(_{77}\)B\(_8\) alloys with \(x = 0, 1, 2\) and 3

Figure 3 compares plots of refined X-ray diffraction data for Pr\(_{15-x}\)Dy\(_x\)Fe\(_{77}\)B\(_8\) alloys with \(x = 0, 1, 2\) and 3. Generally, the X-ray diffraction
The measured X-ray diffraction profiles for all samples look almost similar and are typical of the diffraction profile of the Pr$_2$Fe$_{14}$B phase, referring to the corresponding card number in the Crystallography Open Database (COD: 1008718). However, careful identification of the peak positions for each sample reveals that a small shift due to partial substitution of Dy atoms for Pr in the Pr$_2$Fe$_{14}$B phase can be observed. The results of XRD data analysis indicate that the alloys are not a single phase. Additional phases identified as Pr (COD: 180909) and α-Fe (COD: 5000217) are present in the samples. In addition, the XRD peaks are sharp, indicating a fully crystalline structure that is free from microstrain and fine crystallites. The dominant phases present in the samples are Pr$_2$Fe$_{14}$B and α-Fe. The pure Pr phase appears in addition to the major phase. It is known that regarding the Wyckoff positions, the Nd atom is at sites (4f) and (4g), the Fe atom is at sites (16k1), (16k2), (8j1), (8j2), (4e), and (4c), and the B atom is at the site (4g). In this analysis, the Nd$_2$Fe$_{14}$B structure used was obtained by replacing the Nd atom with Pr atoms at the same site, namely, at sites (4f) and (4g), to produce an atomic coordinate fraction that matches that of the Pr$_2$Fe$_{14}$B structure. Refinement of the X-ray diffraction pattern for all these samples used the atomic coordinate fraction to obtain changes in the structural parameters and structural models of the experimental results. The structural parameter refinement results based on the X-ray diffraction patterns for all Pr$_{(15-x)}$Dy$_x$Fe$_{77}$B$_8$ samples are shown in Table 1.

Referring to the summary of the data analysis in Table 1, it can be seen that as the fraction of the Dy substitution element in the alloy increases, a change in the lattice parameters of Pr$_2$Fe$_{14}$B occurs in which the $a$ and $c$ axis lengths are decreased. The alloys have the same structure of the major phases but with different compositions. In the Pr$_{(15-x)}$Dy$_x$Fe$_{77}$B$_8$ sample, some Pr atoms have been successfully replaced by Dy atoms, which are expected to have an impact on other properties, especially on the magnetic properties of Dy-doped Pr$_2$Fe$_{14}$B.

The energy spectrum of the microanalysis taken from grain morphology of the as-cast samples are shown in Figure 4. The objective is to determine whether Dy-doped Pr$_2$Fe$_{14}$B grains form successfully. The results of elemental analysis measured using energy dispersive spectroscopy (EDS) show that the $x = 0$ sample (Figure 4a) contains the elements Pr, Fe, and Dy, as shown in Figure 4b-d. In detail, the elemental contents in the Pr$_{(15-x)}$Dy$_x$Fe$_{77}$B$_8$ samples are shown in Table 2. Based on the results of this elemental analysis, the phase compositions of all samples are close to the nominal compositions.

Table 1. Summary of identified phases, values of the structural parameters, and the mass fraction of the phases formed in Pr$_{(15-x)}$Dy$_x$Fe$_{77}$B$_8$ alloys.

| Sample | Phase      | Lattice parameter (Å) | Fraction wt.% |
|--------|------------|-----------------------|--------------|
|        |            | $a$       | $b$       | $c$       |              |              |
| $x = 0$| Pr$_2$Fe$_{14}$B | 8.8059(7) | 8.8059(7) | 12.240(1) | 53.52       |
|        | α-Fe       | 2.8662(8) | 2.8662(8) | 2.8662(8) | 45.94       |
|        | Pr         | 3.699(1)  | 3.699(1)  | 10.850(7) | 1.92        |
| $x = 1$| Pr$_2$Fe$_{14}$B | 8.8020(5) | 8.8020(5) | 12.215(1) | 62.94       |
|        | α-Fe       | 2.8657(9) | 2.8657(9) | 2.8657(9) | 36.33       |
|        | Pr         | 3.6988(9) | 3.6988(9) | 10.903(5) | 0.73        |
| $x = 2$| Pr$_2$Fe$_{14}$B | 8.7965(3) | 3.6988(9) | 12.197(6) | 69.98       |
|        | α-Fe       | 2.8671(7) | 2.8671(7) | 2.8671(7) | 29.58       |
|        | Pr         | 3.696(1)  | 3.696(1)  | 10.900(5) | 0.44        |
| $x = 3$| Pr$_2$Fe$_{14}$B | 8.7939(3) | 8.7939(3) | 12.181(6) | 70.60       |
|        | α-Fe       | 2.8664(7) | 2.8664(7) | 2.8664(7) | 29.40       |

Figure 4. The energy spectrum of EDS microanalysis: a) $x = 0$, b) $x = 1$, c) $x = 2$, and d) $x = 3$. 

- **Table 1.** Summary of identified phases, values of the structural parameters, and the mass fraction of the phases formed in Pr$_{(15-x)}$Dy$_x$Fe$_{77}$B$_8$ alloys.
annealed alloys by a permagraph. The external magnetic fields used in the measurement are between -1200 kA/m and 1200 kA/m. This applied magnetic field is significantly inadequate to reach the saturation state. Consequently, the hysteresis loops obtained from this measurement are only minor hysteresis loops. Plots of the second quadrant of the minor hysteresis loop clearly show that the effect of Dy substitution for Pr in the Pr<sub>x</sub>Dy<sub>1-x</sub>Fe<sub>77</sub>B<sub>8</sub> magnetic phase increases the coercivity of the sample as the Dy atomic fraction increases. The increase in the coercivity value is due to the very high anisotropy field value in the RE<sub>2</sub>Fe<sub>14</sub>B phase when the RE is Dy [23]. The coercivity of sample x = 0 is only 140 kA/m and increases to 370 kA/m when x = 3; that is, an increase in the coercivity value of more than twofold occurs. Numerous research reports have shown an increase in coercivity of Pr-Fe-B magnets doped with Dy [24]. Generally, the increase in the magnetic coercivity value with Dy substitution is due to the intrinsic factor and the microstructure of the sample where the grains of the (Pr,Dy)<sub>2</sub>Fe<sub>14</sub>B phase are isolated by the RE-rich grain boundary phase. Finally, the partial substitution of RE atoms with Dy elements in the RE<sub>15</sub>Fe<sub>77</sub>B<sub>8</sub> alloy composition is very efficient for making permanent magnets with extra-high coercivity.

Figure 5b also shows a plot of the hysteresis loops of the as-cast and annealed samples and those that received heat treatment, according to the treatment profile shown in Figure 2. The hysteresis loops do not show hard magnet behaviour at all, instead showing the hysteresis loop of soft magnets. Such hysteresis loops are similar to those derived from Nd<sub>x</sub>Pr<sub>15</sub>Fe<sub>77</sub>B<sub>8</sub> alloys [9], which have been reported by Li Juen et al. [19].

Table 3 summarizes the magnetic properties of all the samples that underwent different heat treatments. The heat-treated samples show a significant coercivity reduction. Phase decomposition occurred after the heat treatment, probably due to the extra-high temperature or possibly due to oxidation. Apart from these two possibilities, an evaluation of the microwave characteristics of the samples using a VNA was carried out.

3.2. Microwave absorption properties of heat-treated Pr<sub>(15-x)</sub>Dy<sub>x</sub>Fe<sub>77</sub>B<sub>8</sub> alloys

The as-cast and annealed alloys exhibit hard magnetic characteristics, as confirmed by the plots of the second quadrant shown in Figure 5a. The coercivity of the alloys as read from such plots is comparable to the typical value of most hard ferrites [25], but the remanence is much higher than 0.19 T, the theoretical remanence of conventional isotropic hard ferrite (assuming that the saturation magnetization value is 0.38 T). Subject to the microstructure and magnetic properties, as reported elsewhere, hard ferrites with reduced coercivity can be applied for radar absorbing materials [26], which show absorption in the x-band frequency (8 GHz–12 GHz). The indicator of the strength of microwave absorption is represented by the reflection loss (RL) parameter [27]. Referring to electromagnetic wave theory [28], it follows that the maximum power absorption will occur when the material input impedance (Z<sub>in</sub>) is equal to the transmission line impedance (Z<sub>0</sub>). The Z<sub>in</sub> reflection coefficient (Γ) and RL are calculated by the following equations:

\[ Z_{in} = \sqrt{\mu_r \varepsilon_r} \tanh \left( \frac{2\pi f d}{c} \sqrt{\mu_r \varepsilon_r} \right) \]  

\[ |\Gamma| = \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \]  

\[ RL(dB) = 20 \log |\Gamma| \]  

|μr and εr are the relative magnetic permeability and electric permittivity of materials, respectively, which are complex numbers. Parameters |
f, d, and c are the frequency, sample thickness, and light velocity, respectively.

The absorption characteristics of a sample may be indicated by the imaginary part of the magnetic permeability since it determines the energy loss in the material when an EM enters the material. The total energy loss of an EM arises from hysteresis loss (which most likely can be neglected due to the relatively weak magnetic field of the EM), eddy current loss (especially in conductive materials), ferromagnetic resonance, which is closely determined by the anisotropy field, and the wave reflection by grain surfaces that weakens the amplitude of the EM. Looking at Eqs. (1) and (2), it becomes clear that the RL value becomes very low when the reflection coefficient (Γ) is low. Theoretically, this takes place when the $Z_{in}/Z_0$ ratio is near or equal to 1. The value of that ratio is set by $\mu_r$ and $\varepsilon_r$. Figure 6 compares the relative magnetic permeability and electric permittivity of as-cast and annealed (Figure 6a) with that of heat-treated Pr$_{15-x}$Dy$_x$Fe$_{77}$B$_8$ alloys (Figure 6b). These plots indicate an increase in the relative magnetic permeability at a frequency of approximately 9.5 GHz for as-cast and annealed samples and at 10.5 GHz for heat-treated samples.

In Figure 7, plots of the RL value in the frequency range of 8 GHz–12 GHz for the as-cast and annealed (Figure 7a) and heat-treated Pr$_{15-x}$Dy$_x$Fe$_{77}$B$_8$ samples (Figure 7b) are presented. The as-cast and annealed samples are characterized by strong absorption with high RL values at a frequency of 10.5 GHz.

The alloy sample with $x = 1$ has the highest RL of -17 dB, which means that more than 85% of the EM entering the sample is absorbed, with the -10 dB bandwidth at 9–12 GHz. The absorption profile looks typical of that of hard ferrite-based absorbers, which have absorption characteristics at radar frequencies. However, rare earth-based absorbers have a large bandwidth. The magnetic properties of the two types of absorbers are similar, in which both are permanent magnets with almost similar coercivities. However, Pr$_{15-x}$Dy$_x$Fe$_{77}$B$_8$ alloys are metal-based absorbers, whereas hard ferrite is a ceramic-based absorber. The heat-treated Pr$_{15-x}$Dy$_x$Fe$_{77}$B$_8$ samples were characterized by broadband absorption with two absorption peaks at frequencies of 9.5 GHz and 11.5 GHz. Consequently, the two absorption peaks almost overlap, leading to very broadband absorption in the whole frequency range (8–12 GHz). Therefore, the RE absorber would be more suitable for operation in the radar frequency range, which could be useful for applications of radar absorbing materials. The absorber with $x = 3$ has absorption peaks with the highest RL value of approximately -15 dB, or more than 82% of the incoming EM can be absorbed at the absorption peak frequencies.

4. Conclusion

Dy-doped Pr-Fe-B alloys with Pr$_{15-x}$Dy$_x$Fe$_{77}$B$_8$ ($x = 0, 1, 2,$ and 3) compositions have been successfully fabricated under melting and successive vacuum annealing. The alloys have two major phases, Pr$_2$Fe$_{14}$B and α-Fe, and a very minor amount of pure Pr, which disappears at $x = 3$. The alloys show ferromagnetic characteristics, with the coercivity value progressively increasing with $x$. In addition, all alloys possess radar absorbing characteristics similar to those of hard ferrite-based absorbers. The heat treatment applied to the as-annealed samples changed the properties from those of a permanent magnet to those of a broadband
RAM with two absorption peaks that almost overlap, leading to very broadband absorption in the whole x-band frequency range. The absorber with \( x = 3 \) has absorption peaks with the highest RL value of approximately \(-15\,\text{dB}\), or more than 82\% of the incoming microwave can be absorbed at the absorption peak frequencies.

**Declarations**

**Author contribution statement**

Nanang Sudrajat: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Yana Taryana: Performed the experiments; Analyzed and interpreted the data.

Dedi Dedi: Analyzed and interpreted the data; Contributed reagents and materials, analysis tools or data.

Wisman Ari Adi: Analyzed and interpreted the data; Wrote the paper.

Lucky Darmawan: Performed the experiments; Contributed reagents and materials, analysis tools or data.

Azwar Manaf: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents and materials, analysis tools or data; Wrote the paper.

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**Data availability statement**

Data included in article/supplementary material/referenced in article.

**Declaration of interests statement**

The authors declare no conflict of interest.

**Additional information**

No additional information is available for this paper.

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