Development of motor design technologies using high performance magnets

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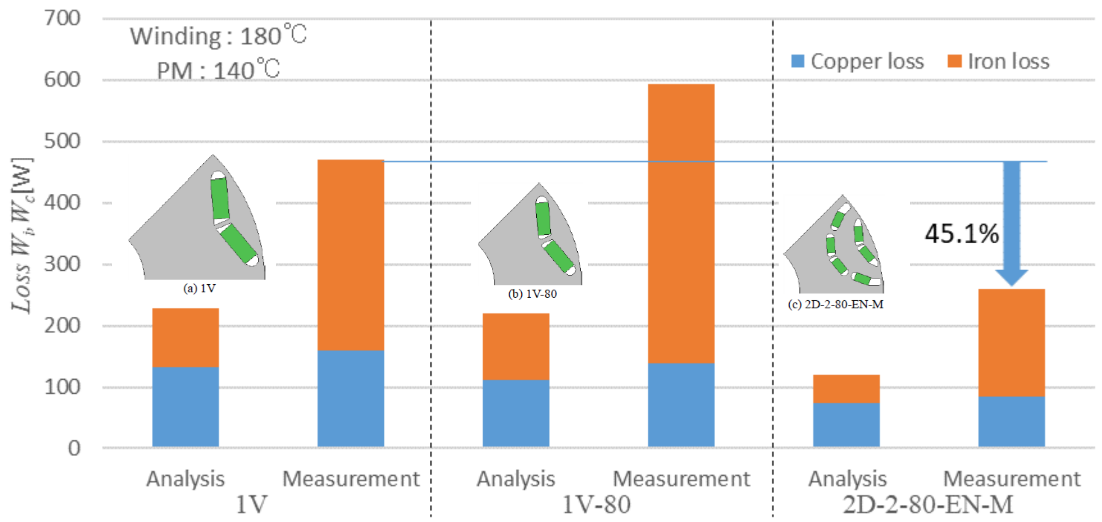
The Technology Research Association of Magnetic Materials for High-Efficiency Motors (MagHEM) was founded in 2012 to develop the innovative high-performance magnets without/less rare-earth materials which exceed current magnets with rare-earth materials in performance, the high-efficiency soft magnetic materials (Iron core) for internal loss reduction, and compact high-efficiency motors.

Targets of R&D are new magnets exceeding Neodymium magnets with 2 times in (BH)max (180℃), and high efficiency motors with 40% reduction in loss, 40% improvement in power density using new magnets.

We have achieved the above target in simulation[1][2][3]. In this paper, we compare the characteristics of a small-diameter V-shaped magnet arrangement prototype(1V-80) and a small-diameter double-layered arranged prototype(2D-2-80-EN-M) with a conventional single-layered V-shaped arranged prototype(1V) by actual machine measuring.

Then we compared measured data to analysis data. At a result measured loss for the 2D-2-80-EN-M prototype was reduced by more than 40% compared to that for the 1V prototype as well as the analysis data[4].



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Recent advancement of permanent magnet materials developments for vehicle electrification and expectation for future research.

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1.Background

Recently, vehicle electrification expands rapidly. It is well known that electrified vehicle has additional component compare to conventional gasoline vehicle, i.e. battery, inverter and electric motor. At this moment, supply and demand seems to be acceptable for all additional electric component. However, most of future forecast says that amount of electrified vehicle become two to five times larger than current vehicle sales. This means that we need number of electrified unit, at least, more than two times compare to current demand. For example, IEA scenario described in Energy Technology Perspective 2017 forecasts electrified vehicle increase from 14 million in 2020 to 40 million in 2030 [1]. When we look at even only around vehicle technological shift, it is easy to forecast enormous number of rare-earth magnet will be needed. From this circumstance, we are researching coerciviy mechanism of rare-earth magnet and consider what we can do for balancing global supply and demand of rare-earth materials.

Beside vehicle application, future demand of magnet may rise not only from vehicle electrification but from mobility for short commute, logistics, robotics etc. When we include these application for future forecast of rare earth demand, larger amount of rare earth demand is expected. In order to get over this problem, we should look at wider magnet composition range. In this talk, I will present recent progress of magnet R&D in TMC based on data driven approach.

2.Current situation of magnet research in TMC

Regarding $RE_2TM_{14}B$ compound, we adopt informatics technique for accelerating magnet research. For designing performance of magnet, we need not only magnetic properties of compound but microstructural control technique. From research achievement in the past, we already aware that coercivity can be enhanced by surface modification. It is well known that surface modification consists of two parts, one is compositional control of grain shell [2,3,4] and recovering surface distorted region [5]. In order to control these two parts, we can use grain boundary diffusion technique and low temperature annealing. On the other hand, compositional dependence of magnetic properties, many reports were published in the past, but information had not stored systematically. To get over this situation, we are now producing data space for designing magnet [6]. When we look only at room temperature, it seems that there is no room to enhance magnet performance in $RE_2TM_{14}B$ system. Combining experimental data, empirical model and first principle calculation, our group expand data space, about 100 data from experiment and several thousand data from first principle. Mining established data space, we can find better performance potential composition range in elevated temperature. Currently, we are strongly push forward data driven research. To utilize all information contained in all data, e.g. XRD, SAS, micro-scope image etc., we develop data extraction technique utilizing informatics algorithms and integrate these achievements into cloud server system.

3. Future perspective

In near future, many kind of electro-magnetic devices become important for future application not only for vehicle electrification but for various application. In order to Designing magnetic performance suited for each application, magnetic properties data space will take important role, I think. I hope “Data driven material research” will evolve “Data driven material design and development”. To achieve this context, culture for storing research data will become more important.

Acknowledgement
I would like to thank to research collaborators of TMC's magnet research activity, Prof. Miyake's group AIST and Prof Fukushima's group ISSP. I deeply thank to Dr. Harashima, Dr. Doi and Dr. Matsumoto for executing first principle calculation and machine learning. This work was partly supported by the future pioneering program Development of Magnetic Material Technology for High-efficiency Motors commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

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T(K)

Development of high coercivity Nd-Fe-B permanent magnets with improved thermal stability

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(Nd,HRE)-Fe-B based permanent magnets (HRE : heavy rare-earth) are utilized in the traction motors of (hybrid) electric vehicles which are resistive against demagnetization at the operating temperatures up to 200°C. However, due to the limited natural resources of HRE elements such as Dy and Tb, the development of HRE-free Nd-Fe-B based magnets with sufficiently large coercivity is needed. To meet this demand, the improvement of the thermal stability of coercivity is desired. It is well known that the temperature dependence of coercivity in commercial Nd-Fe-B sintered magnets deviates from temperature dependence of the anisotropy field of $Nd_2Fe_{14}B$ (Fig. 1a). In this study, we investigated the mechanism of pronounced thermal degradation of coercivity of Nd-Fe-B magnets by combining micromagnetic simulations, magnetic domain observations, and multi-scale microstructure characterizations using scanning transmission electron microscopy and atom probe tomography [1-6].

In this talk, we first discuss the reason for the poor thermal stability of coercivity in Nd-Fe-B based permanent magnets. In previous micromagnetic simulation studies, the temperature dependent magnetization of intergranular phase has not been considered. In this work, we found that the concavity of H_c-T can be reproduced by incorporating the temperature dependent magnetization of ferromagnetic intergranular phase (Fig. 1b) [6]. Decrease of saturation magnetization and Curie temperature of the grain boundary phase were found to be crucial to improve the thermally stability of the coercivity of Nd-Fe-B magnets. Based on this simulation results, we demonstrated excellent hard magnetic properties of μ_0H_c=2.5 T, μ_0M_r=1.4 T, and an excellent thermal stability of coercivity of -0.33 %/°C by infiltrating low-melting-temperature Nd-HRE-Cu alloy into hot-deformed Nd-Fe-B magnets (Fig. 2). Based on the microstructure studies and micromagnetic simulations, we will discuss how the formation of non-ferromagnetic grain boundary phase and HRE-rich shell are beneficial to achieve high coercivity with excellent thermal stability (Fig. 2).

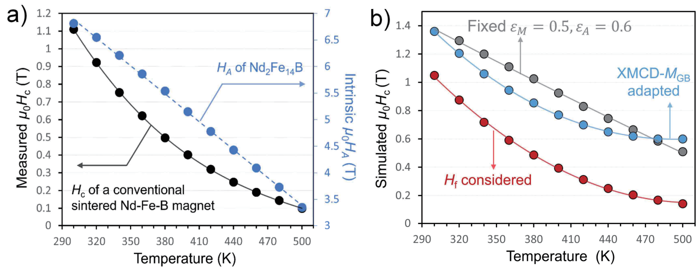

Fig. 1 (a) Comparison of the temperature dependence of coercivity of conventional Nd-Fe-B sintered magnet and intrinsic anisotropy field of $Nd_2Fe_{14}B$ [6]. (b) Simulated temperature dependence of coercivity when grain boundary is ferromagnetic with and without consideration of temperature dependence of M_s^{GB}, A^{GB}, and thermal activation [6].

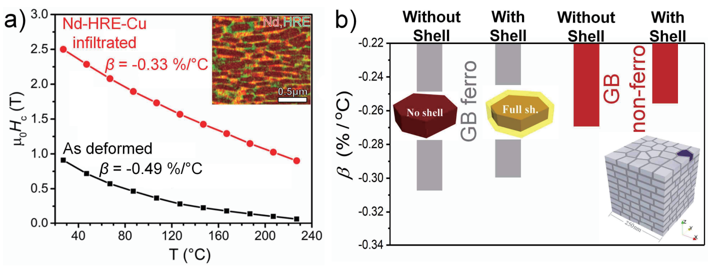

Fig. 2 Coercivity as a function of temperature obtained from as hot-deformed and Nd-HRE-Cu infiltrated magnet. STEM-EDS map of Nd and HRE is shown in inset. (b) Simulated temperature coefficient of coercivity for the hot-deformed model with $Nd_2Fe_{14}B$ grains covered with and without HRE-rich shell and different GB magnetism [5].

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Computational thermodynamics and microstructure simulations applied to grain boundary engineering in Nd-Fe-B sintered magnet

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To control the coercivity of the Nd hard magnet efficiently, we should understand the thermal stability of constituent phases and the microstructure changes observed in the hard magnets during their thermal processes. Since the CALPHAD method and the phase-field method have been recognized as promising approaches to realize the phase stability and microstructure developments in the engineering materials recently,[1)] we applied these methods for understanding the nature of the grain boundary phase and the microstructure developments in Nd-Fe-B hard magnet.

Figure 1 demonstrates the two-dimensional simulation result on the microstructure changes of Fe-15.3 at %Nd-5 at %B-0.2 at %Cu alloy with isothermal aging at 873K. Upper and lower figures are the phase field and the composition field, respectively. The white, black and gray parts in the phase-field are the $Nd_2Fe_{14}B$ phase (T_1 phase), the liquid phase, and the Nd solid phase, respectively, and each number indicated by *t* ' is a dimension less aging time. The degree of red color in the composition field means the local Nd concentration in the microstructure. At early stage, the Nd solid phase starts dissolving, and a liquid phase appears at grain boundary region. With aging, the Nd solid phase gradually disappears, and the Nd-rich liquid phase penetrates along grain boundary region, then the characteristic morphology of microstructure that the T_1 grains are uniformly covered with thin film of liquid phase appears. It has been elucidated experimentally that the Cu addition lowers the melting point of the liquid phase, because a eutectic reaction exists in the Cu-Nd binary phase diagram. By increasing the thermodynamic stability of liquid phase by Cu addition, the volume fraction of the liquid phase also increases, and then, it can be understood that the characteristic morphology is stabilized. Furthermore, when we focused on the final composition field carefully, the brightness of red color at the tri-junction region of T_1 grains differs from that at the grain boundary region between T_1 grains. This is because of the phase separation in the liquid phase, i.e., $L \rightarrow L_1 + L_2$. Since a phase separation of liquid phase has been reported in the calculation of the Fe-Cu-Nd phase diagram, the liquid phase separation induced by Cu addition is not an unusual phenomenon. When we imagine the coarsening process of the liquid phases, the L_1 phase should move over the L_2 phase; in other words, the movements of the L_1 and L_2 phases will interfere with one another during coarsening. The phase separation of liquid phase can contribute to stabilize the characteristic morphology (uniform coating of the T_1 grains by the liquid phase) temporally.

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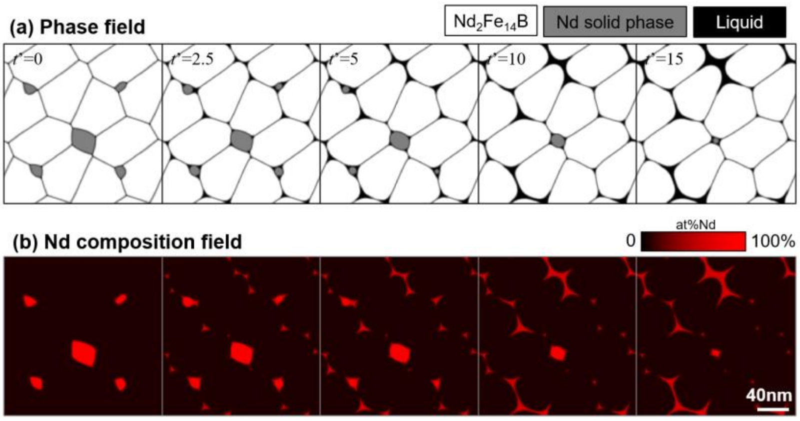

Fig.1 Phase-field simulation of the microstructure changes in Fe-15.3 at %Nd-5 at %B-0.2 at %Cu alloy with isothermal aging at 873K.

Acknowledgement: This work was partly supported by the project in Elements Strategy Initiative Center for Magnetic Materials (ESICMM) founded by Ministry of Education, Culture, Sports, Science and Technology, Japan.

Determination of constituent phase changes in Nd-Fe-B-Cu sintered magnets on heating and cooling processes by *in-situ* synchrotron X-ray diffraction

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For the production of Nd-Fe-B sintered magnets with high coercivity, optimized heat treatments to improve the microstructure are essential. Thus, the clarification of the thermodynamic behavior of secondary phases that form microstructure is important to manufacture such magnets effectively. To clarify phase changes in bulk magnets during heating treatments, we have conducted *in-situ* high-temperature synchrotron X-ray diffraction (XRD). The previous measurements for Nd-Fe-B-Cu bulk sintered magnets were successfully conducted only on heating [1,2]; however, it is difficult to distinguish between reversible and irreversible changes with temperature, which is essential for comparing experimental and theoretical results. Therefore, it is desirable to observe phase changes on cooling as well, although it is challenging because of the easy oxidation of rare-earth elements at high temperatures. In this study, we improved the experimental setup in the high-temperature *in-situ* XRD and carried out measurements on both heating and cooling.

A rectangular rod-shaped isotropic as-sintered magnet with the composition of $Nd_{13.74}Fe_{78.35}B_{5.92}Cu_{0.10}O_{1.88}$ in at.% was used. The preparation method has been reported elsewhere [2]. Synchrotron XRD measurements were conducted using a carefully designed sample holder to prevent the oxidization of magnets during heating. Synchrotron XRD profiles were collected using a high-resolution one-dimensional solid-state detector at the BL02B2 beamline of SPring-8. The sample was heated from room temperature to 1100°C using a cylindrical heater. The experimental results were compared to the computational phase diagram of this magnet based on the combined *ab initio*/CALPHAD approach [3].

We have observed similar XRD profiles at the same temperatures on heating and cooling except for slight differences in peak intensities, indicating the successful observation of the almost reversible phase changes. Figure 1 exhibits the temperature dependence of the amounts of secondary phases on cooling. There are two remarkable changes, which are compared to the computational diagram. One is the change in the amount of dhcp-Nd phase between 500°C and 650°C, which is similar to the previous observation except for the slight difference in temperatures [1,2]. This phase change is considered to originate from the eutectic reaction of dhcp-Nd and NdCu phases, as confirmed by the calculation. The NdCu binary phases, which were suggested in previous reports [3,4], were not detected in XRD probably because of the broad XRD peaks resulting from small crystalline sizes on the order of nanometers. The other finding is the change in the amounts of fcc-NdO_x and hcp-Nd_2O_3 above 1000°C. The result is likely explained as follows: fcc-NdO_x ↔ hcp-Nd_2O_3 + Liquid (the rightward and leftward reactions represent a phase change on heating and cooling, respectively). Although the phase change temperature we observed is much higher than that in the calculated phase diagram of this magnet composition (650°C), the temperature is close to that in the calculated NdO binary diagram (1100°C) [4]. This means that the fcc-NdO_x phase does not show the phase equilibrium with the other secondary phases in the Nd-Fe-B-Cu sintered magnet but shows the phase change almost independently.

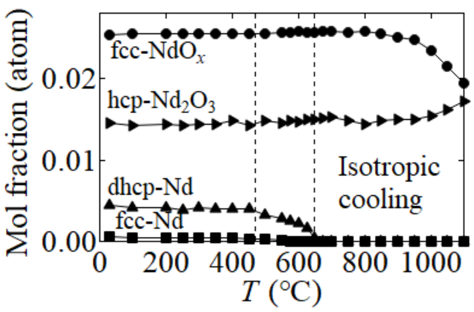

Fig. 1 Temperature dependence of amounts of secondary phases in the Nd-Fe-B-Cu isotropic sintered magnet on cooling.

We thank T. Nishiuchi and T. Fukagawa of Hitachi Metals, Ltd. for supplying the samples. Part of this work is supported by ESICMM under the outsourcing project of MEXT (JPMXP0112101004).

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Development of high performance anisotropic magnetic powders for bonded magnets

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The preparation of high-performance anisotropic magnetic powder is the key to obtain high-performance bonded magnets. In this talk, I will report the research results of our group on anisotropic permanent magnetic powders and magnets. By using neutron diffraction, magnetic measurement, electron microscopy, and other techniques combined with the electronic structure calculation, the relationship between the structure and magnetic properties of magnetic materials was investigated. The physical roots of the interstitial atom effect to improve the magnetic properties of the material are clarified, and the preparation of defect-free single-crystal-like particles is proposed to synthesize high-performance magnets powders. Based on the technical route of anisotropic permanent magnetic powder, the key technologies and equipments for the industrialization of high-performance anisotropic $Sm_2Fe_{17}N_x$ and $Nd(Fe,M)_{12}N$ magnetic materials and magnets have been developed. We also investigated the critical mechanism of the formation of textured NdFeB and MnBi permanent magnetic powder and the methods to achieve high coercivity and high maximum energy product. The high-performance magnetic powders with high-temperature stability were obtained. Finally, we explored the synthesized hybrid bonded magnets based on these magnetic powders, which is critical to realizing the mass production of anisotropic bonded magnets.

Sm-Fe-N powders and bulk magnets by ultra-low oxygen processes

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Many researchers have tried an anisotropic $Sm_2Fe_{17}N_3$ sintered magnet so far in order to obtain a heat-resistant permanent magnet with high coercivity and remanence. However, it had been stagnating for a long time because of two obstacles: one is thermal decomposition which limits the sintering temperature, and the other is significant deterioration of coercivity during sintering. For the latter problem, we have so far clarified the involvement of surface oxide film on the raw powder, and then demonstrated that powders with less surface oxide can suppress the coercivity deterioration [1). Based on these facts, we built up our own low-oxide powder metallurgy system to produce a sintered magnet without air-exposure from pulverization to sintering. Using this process, the present study examined the possibility of creating a high-performance $Sm_2Fe_{17}N_3$ sintered magnet [2)]. Our system consists of some glove boxes connected in series, and these glove boxes are equipped with functions such as pulverization, magnetic alignment, sintering, and so on.

First, the $Sm_2Fe_{17}N_3$ fine powders with less surface oxidation were produced by using a high-pressure jet-mill under the low oxygen environment. By varying milling conditions, fine powders of various mean sizes down to 1 micrometer could be prepared. Among these, the finest powders had the largest coercivity more than 14 kOe but their $(BH)_{max}$ was very low due to kink and powder agglomeration. The powders prepared with appropriate conditions exhibited the large $(BH)_{max}$ of 43 MGOe as well as low oxide less than one third of conventional.

The prepared powders were then subjected to magnetic-aligned compaction and rapid sintering under the low oxygen atmosphere. As expected, the powders with less oxide film showed only slight decrease in coercivity of less than 15% by sintering, whereas the conventional techniques suffered the reduction more than 70% [3)]. Sintered density was reached to a relatively high value of 91% by even sintering temperature of 500 ºC. On the other hand, the achievement of suppression of coercivity deterioration has revealed a new problem of decrease in saturation magnetization. Specifically, the saturation magnetization of powder was reduced by about 5% during sintering. This reduction would be derived from low crystallinity of the jet-milled powder. Due to this reduction in saturation magnetization, $(BH)_{max}$ of sintered magnets was 24 MGOe regardless of the high $(BH)_{max}$ of raw powder. Nevertheless, this value was achieved for the first time by the accessible sintering technique.

We are also conducting the research to improve the coercivity of sintered magnets by direct metal coating on $Sm_2Fe_{17}N_3$ particles without surface oxide film. In the neodymium magnets, it is known that the coercivity is greatly improved by adding a specific other element into the grain boundaries. Hence, there is a possibility that the same effect can be obtained in $Sm_2Fe_{17}N_3$. Currently, we discovered that several elements such as Al, Ce, Ru, Mn, Ti and so on, are effective for improving the coercivity of $Sm_2Fe_{17}N_3$.sintered magnet besides Zn which has been ever known to increase the coercivity [4)]. In addition to the low oxygen powder metallurgy technique, we are trying to prepare $Sm_2Fe_{17}N_3$ nanoparticles by a low-oxygen thermal plasma synthesis. Nanoparticles are expected to improve not only coercivity but also sinterability. We have so far confirmed that this synthesis method is able to synthesize Sm-Co nanoparticles.

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Synthesis of R-TM hard magnetic powder by thermal plasma

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[Background]

Not so many compounds with higher potentials than those of $Nd_2F_{14}B$ as a permanent magnet material have been reported[1, 2]. Among them, only compounds that have been successfully synthesized in bulk are Ti-less compounds with a $ThMn_{12}$ structure[3] or nitrides with a $TbCu_7$ structure[4]. However, there are some difficulties to obtain an anisotropic fine powder of these compounds. It is well known that single-crystalline for an anisotropic magnet with higher remanence and fine particles for higher coercivity are necessary to exploit the potential of the compound as a permanent magnet. Here, the induction thermal plasma (ITP) process as a new process for further particle size refinement was focused on. For nanopowder fabrication, this process has the advantages of a high production rate, control over particle size, and an inherently contamination-free process. Moreover, it could be possible to obtain fine particles with stable and metastable phases by tuning the cooling rate. Attention should be paid to the handling of ultrafine metal particles which is highly reactive with oxygen and humidity in the air. Recently, we developed a low-oxygen ITP system (LO-ITP)[5], which enables us to prepare ultrafine metal powder in the controlled low-oxygen atmosphere. This technique was applied to prepare single-crystal ultrafine R-Fe alloy powders, especially for R = Nd in this research, with stable and metastable phases.

[Experiment]

The mixed powder of Fe (Kojundo Chemical Lab. Co., Ltd., Japan) and Nd with the atomic ratio of Nd : Fe = 2 : 3 was used as a starting powder. A TP-40020NPS (JEOL Co., Ltd.) was used for the ITP process, and a TP-99010FDR (JEOL Co., Ltd.) was used as the powder feeding system. In the thermal plasma process, the conditions of a process pressure of 100 kPa and a feed rate of up to 0.3 g/min were used. Transmission electron microscopy (TEM) sample was prepared by focused ion beam (FEI, Scios), and the microstructure was observed by TEM (JEOL ltd., JEM-ARM200CF equipped with CEOS ASCOR corrector) using an accelerating voltage of 120 kV. The anisotropic powder samples were prepared in the external magnetic field of 9 T at 400 K.

[Result and discussion]

According to the XRD profile, Nd-Fe alloy particles with the stable Th_2Zn_{17}-type phase and the metastable $TbCu_7$-type phase were selectively prepared by controlling the cooling rate of the ITP process. The single-crystal of particles was confirmed by HAADF-STEM. The fine particle size of less than 100 nm was obtained. From the numerical calculation, it was found that the alloying mechanism revealed that Nd and Fe nucleate and condense simultaneously in the liquid temperature range due to the formation of the alloy droplet during cooling. Both obtained powders could be aligned by the external magnetic field, indicating that obtained ultrafine powders by the LO-ITP process were anisotropic, which was confirmed by the XRD and magnetic measurement. Therefore, this process is a new promising way to achieve a new-generation anisotropic permanent magnet.

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