doi:10.1017/S143192761300648X
Microsc. Microanal. 19 (Suppl 2), 2013

Applications of the Hybrid Ion Milling Method to Neodymium Magnets

Takashi Shidara[1], Mami Konomi[1] , Syunya Watanabe[1] , Hirobumi Muto[2] and Hisayuki Takasu[2]

[1.] Global Application Center, Hitachi High-Technologies Corp., KANAGAWA SCIENCEPARK, R&D BUSINESSPARK BLDG C-1F, 3-2-1, Sakado, Takatsu, Kawasaki, Kanagawa 213-0012, Japan
[2.] Advanced Microscope System Design Department, Hitachi High-Technologies Corp., 882, Ichige, Hitachinaka, Ibaraki, 312-8504, Japan
E-mail: shidara-takashi@naka.hitachi-hitec.com

Neodymium magnets are widely utilized in various products such as hard disk driver and motors of hybrid cars and their magnetic characteristics are related to organizational structure, composition distribution and crystal orientation. To investigate the characteristics of Nd magnets, the cross sectional observations of Nd magnets using a SEM are reported [1]. In general, mechanical polishing is useful to reveal wide cross-sections. However, mechanical polishing methods can often induce mechanical artifact in samples that contain various materials such as structural and constituent changes, oxidation and exfoliation. The Nd magnet is commonly known for cracks developing in the grain boundary that are induced by oxidization. The ion milling method is useful for producing a wide cross-section of the composite sample. In a previous study, the surface of Nd magnets processed by ion milling, warped. Next, we have optimized the milling conditions of dross-section and alternately used flat milling as a two step process to prepare the cross sections of Nd magnets using a Hitachi ion milling system, model IM4000. We observed their fine structures using a FE-SEM and easily analyzed using the EDX system.

Figure 1 showed BSE images of the Nd magnet prepared by the hybrid ion milling method. In figure 1a, many striations (arrow head) by Ar ion beam were widely seen. After flat milling with a low irradiation angle of Ar ion beam, the striations were removed as shown in figure 1b. To obtain flatter surface, the sample stage were slightly swung and the smooth surface were finally revealed indicated in figure 1c. Figure 2 showed the BSE images of the whole cross section of the Nd magnet prepared by the hybrid milling method. At a low magnification, the whole cross-section of approximately 1 mm in diameter was indicated (figure 2a). In figure 2b, Ni plating for anti-oxidization in the outer layer and other phases, which are $Nd_2Fe_{14}B$ compound phase, Nd-rich phase, and B-rich phase, were observed. Figure 2c clearly showed that Nd-rich phase were co-placed around $Nd_2Fe_{14}B$ grain. It is indicated that the coercive force of Nd magnets depends upon its crystal grain boundary and smaller graina have stronger coercive force, and/or Nd-rich phase co-placed around the grain. The hybrid milling method is a useful technique to realize $Nd_2Fe_{14}B$ grain size and the distribution of Nd-rich phase. Figure 3 showed the results of EDX analysis of the same sample shown in figure 1c. As the results, the distributions of the three phases were well correlated with BSE images. The B-rich phases were heterogeneously distributed in various boundaries. This study emphasized that the hybrid milling by the IM4000 was useful to observe and analyze wider and flatter cross sections of Nd magnets.

References

[1] M. Sagawa, *et al*, Neozimu Zisyaku No Subete(All of the neodymium magnet) p. 103.(2011)

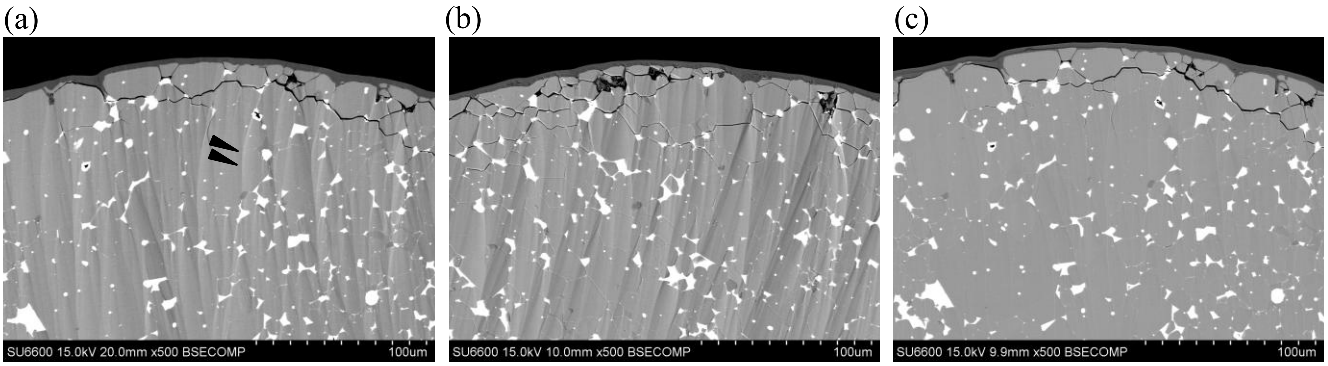


Figure 1. BSE Images of Nd magnets prepared by cross section milling (a), additional flat milling with sample rotation (b) and with sample swing (c). Instrument: SU6600 FE-SEM, Acc. Volt. 15 kV, Magnification: x 500.Sample Prep. : IM4000

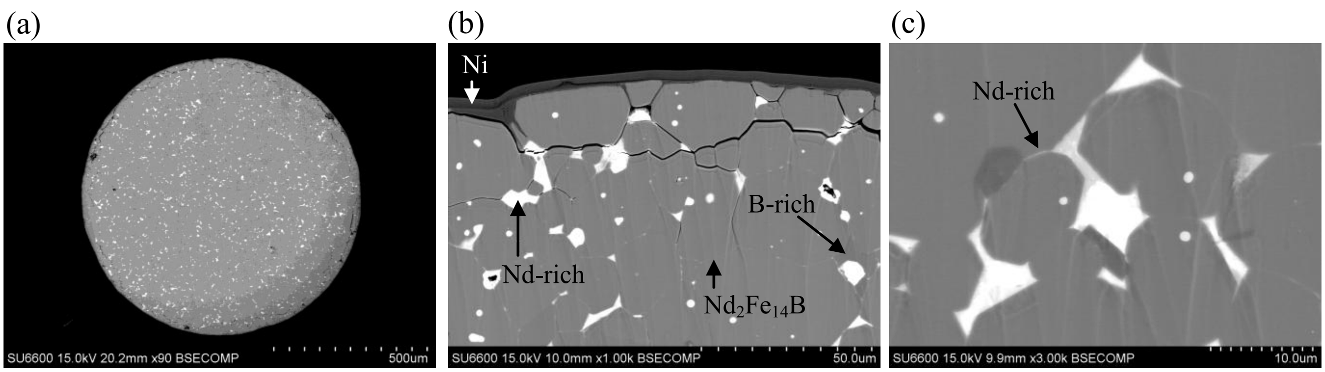
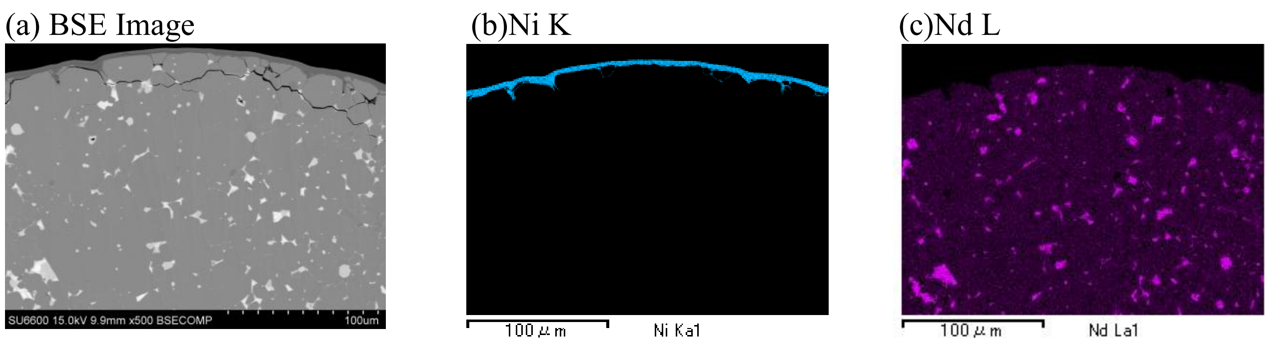


Figure 2. BSE Images of the neodymium magnet (a) x 90, (b) x 1k, (c) x 3k, Instrument: SU6600 FE-SEM, Acc. Volt. 15 kV, Sample Prep.: IM4000



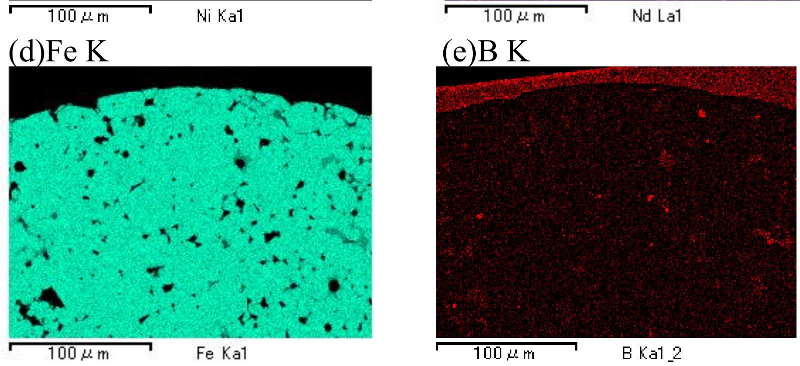


Figure 3. BSE and EDX mapping Images of the neodymium magnet Observation: SU6600 FE-SEM, Acc.Volt.: 15 kV, Mag.:x 500, Sample Prep.: IM4000

https://doi.org/10.1017/S143192761300648X Published online by Cambridge University Press