In-situ X-ray diffraction study of crystallization process of Nd-Fe-B amorphous alloys under high pressure

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Attempts to obtain the next generation high-performance permanent magnets have opened up a theoretical possibility of surpassing $Nd_2Fe_{14}B$ in magnetic performance by preparation of a nanocomposite ferromagnetic material in which a ferromagnetic phase with a large saturation magnetization is magnetically polarized by intergranular exchange interactions with surrounding hard magnetic phases[1]. In order to study possibility of manipulating microstructure and improving magnetic properties by applying a large pressure, crystallization process of amorphous precursors of $Fe_3B/Nd_2Fe_{14}B$ nanocomposite permanent magnets has been investigated *in-situ* using a multi-anvil press and a X-ray diffractometer installed at BL04-B1.

Melt-spun amorphous flakes of $Nd_5Fe_{77}B_{18}$ and $Nd_5Fe_{74}Cr_3B_{18}$ were subject to pressure of 6 GPa produced in BN cells installed in an MgO block and heated stepwise up to 800 ℃. Cr-doped formula was selected because of a prominent effect of Cr on phase selection during crystallization[2]. X-ray energy spectra at a diffraction angle of five degrees were recorded in an accumulation period of 150 seconds at each temperature, which was followed by step-wise raise of temperature. Overall average heating rate was approximately three degrees per minute.

Figure 1 shows X-ray diffraction patterns of $Nd_5Fe_{77}B_{18}$ obtained at 590, 620, and 670 ℃. Hallow patterns typical for an amorphous metal were clearly observed for both compositions at temperatures below 590 degrees Celsius. Slight changes in the diffraction pattern began to occur at 600 and 610 degrees Celsius, respectively, for $Nd_5Fe_{77}B_{18}$ and $Nd_5Fe_{74}Cr_3B_{18}$. These are regarded as onset temperatures of crystallization. Completion of crystallization, which was determined from disappearance of the hallow pattern, was observed when temperature was raised to 670 and 660 ℃, respectively. Phases present in this stage were suggested to be Fe_3B, $Nd_2Fe_{14}B$, and α-Fe, although a clear identification was not possible due to mutual overlap of diffraction lines of these phases. Application of a high pressure seems to cause stabilization of $Fe_3B/Nd_2Fe_{14}B$, which was not formed in the previous ambient-pressure crystallization experiment on $Nd_5Fe_{77}B_{18}$[2].

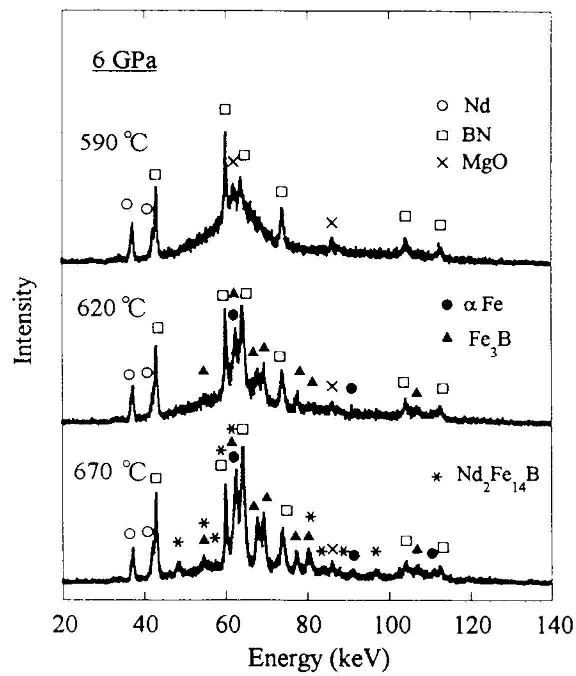

Fig. 1 X-ray diffraction patterns of $Nd_5Fe_{77}B_{18}$ at 590(top), 620(middle), and 670(bottom) ℃ under 6 GPa pressure

In conclusion, in-situ X-ray diffraction analysis of crystallization process of Nd-Fe-B amorphous alloys was successfully performed using the high-pressure-high-temperature apparatus installed at BL04-B1. In order to clarify the effect of pressure, isothermal in-situ observation may be helpful. This study has proved that such observations are technically possible using the same apparatus.

References

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