# More accurate hysteresis curve measurement in large Nd-Fe-B sintered magnets at elevated temperatures

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The hysteresis curve for large Nd-Fe-B sintered magnets has usually been obtained through the hysteresis graph (HG) method. Recently, widespread discussion has addressed the accuracy limit of hysteresis curve measurement for large Nd-Fe-B sintered magnets at elevated temperatures. The abnormality of magnetization ($J$) on the hysteresis curve is known to directly affect squareness measurements, such as the differential susceptibility ($dJ/dH$) near $H_{cJ}$ and $H_k/H_{cJ}$, in the HG method for Nd-Fe-B sintered magnets with higher coercivity ($H_{cJ}$) at room temperature, where $H$ and $H_k$ are the magnetic and knee fields, respectively. The abnormality of $J$ is caused by the distortion of magnetic flux distribution around the sample at high fields. To obtain a more accurate hysteresis curve for large Nd-Fe-B sintered magnets at elevated temperatures, we employed a superconducting magnet-based vibrating sample magnetometer (SCM-VSM) with a maximum magnetic field ($H_m$) of 8 MA/m. The values of $dJ/dH$ near $H_{cJ}$ and $H_k/H_{cJ}$ obtained from the SCM-VSM were compared with those obtained from the HG method at 298 to 473 K. The HG method employed a large electromagnet ($H_m$ of 2.7 MA/m) with improved fixture of heated Fe-Co pole tips. The values of $H_{cJ}$ for the Nd-Fe-B sintered magnet samples A and B were 1.2 and 2.7 MA/m, respectively. The compositions of samples A and B were $Nd_{10.4}Pr_{3.0}Dy_{0.4}Fe_{bal.}Co_{0.6}Al_{0.6}B_{6.1}$ and $Nd_{9.5}Dy_{4.1}Fe_{bal.}Co_{0.6}Al_{0.5}B_{5.8}$, respectively. Cylindrical and spherical samples were machined from the same sintered block for the HG and SCM-VSM measurements, respectively. The cylindrical samples used for the HG method had a diameter ($D$) of 10 mm and lengths ($L$) of 7, 14, and 21 mm. A spherical sample with a $D$ of 7 mm was prepared using the two-pipe method for the SCM-VSM measurement. To remove the deteriorated surface layer of approximately 8 μm, all processed samples were chemically etched in 3% $HNO_3$ solution for 1 min.

In the results obtained from the SCM-VSM and HG (in all cases of $L/D$) measurements, the values of $dJ/dH$ near $H_{cJ}$ increased gradually as the temperature rose for sample A, which had a lower $H_{cJ}$. The differences between the values obtained from these methods were small. In contrast, the values of $dJ/dH$ near $H_{cJ}$ obtained from the HG method (in all cases of $L/D$) increased rapidly as the temperature rose for sample B, which had a higher $H_{cJ}$, as shown in Fig. 1. The values obtained from the SCM-VSM method gradually decreased as the temperature rose. However, the measured values from the two different methods generally agreed well at temperatures above 423 K, where $H_{cJ}$ was reduced below 1.2 MA/m. The values of $H_k/H_{cJ}$ obtained from the SCM-VSM method at 473 K were superior to those obtained from the HG method (in all cases of $L/D$) regardless of $H_{cJ}$ (Fig. 2). Greater $L/D$ led to easier uniform magnetization of cylindrical magnets, causing larger $L/D$ to improve $dJ/dH$ near $H_{cJ}$ and $H_k/H_{cJ}$ measurements with the HG method.

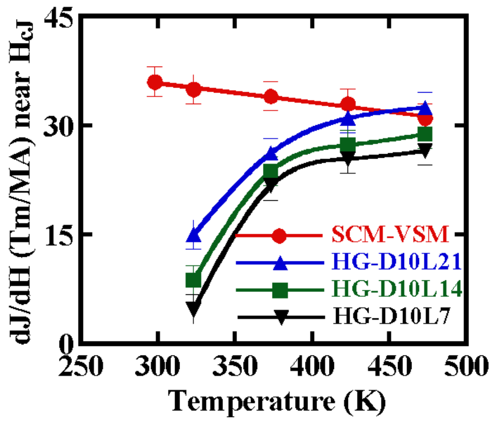


Fig.1 Temperature dependence of dJ/dH near $H_{cJ}$ measured by SCM-VSM and HG methods for Sample B.

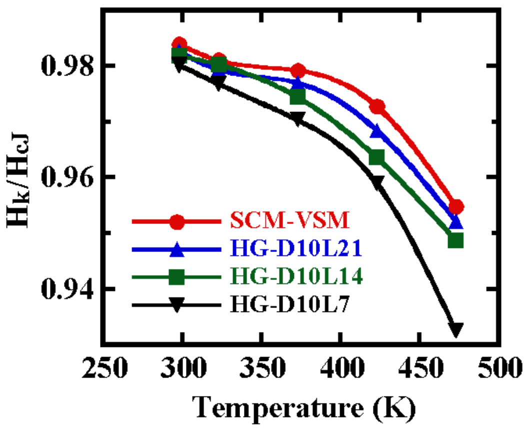


Fig.2 Temperature dependence of $H_k/H_{cJ}$ measured by SCM-VSM and HG methods for Sample A.