Chapter IV

Experimental Details

The aim of this study was to investigate the effect of nitrogen absorption on the magnetic properties of Sm_nCo_m compound. We assumed that this insertion did not effect to the crystal structure of a host compounds. The amount of nitrogen absorbed was controlled by the use of different annealed temperatures. We started with powder form of $SmCo_5$, Sm_2Co_7 and Sm_2Co_{17} obtained from Yue Long Non-Ferrous Metals Ltd (China), Johnson Metthey and Leico Industries (U.S.A.). Sample preparation strongly influenced the magnetic properties, especially on the macroscopic properties e.g. coercive force and the energy product. However, in this investigation we were only interested in one of the microscopic properties, the magnetization.

The sample, powder of Sm-Co compounds in three ratios, were weighted precisely by the Strorius balance. They were place into the aluminum oxide crucible and annealed under nitrogen atmosphere in an enclosed chamber in Linberg/Bluem furnace, figure 4.1. The flow rate of nitrogen into the chamber was 6 cubic feet per hours. Changing the annealing temperature with fixed annealed time was used to control the amount of nitrogen absorption. The selected temperatures was in the range 200-400 ^{o}C. This range was used since samarium evaporate at a high temperature, which leads to the transition of the compounds to the formation Co-rich compounds, as has been reported in $Sm_2Fe_{17}N_x$ compound heated at 550-800^{o}C annealed temperature range (Ding et al., 1992).

An other reason was the difference in absorption range of RE-Fe and RE-Co compounds. Katter et al. (1992) reported on the absorption range of Sm_2Fe_{17} and

Sm_2Co_{17} in N_2 and air. The former absorbed N_2 from 550-800 K or 300-600 °C. The later absorbed nitrogen in the range 500-870 K or 250-650 °C, as seen in figure 4.2. We assumed that $SmCo_5$ and Sm_2Co_7 absorb N_2 in similar range. After annealing, the specimens were weighed again to determine the mole fraction of nitrogen, assuming that all of Sm_nCo_m become to $Sm_nCo_mN_x$ compounds. The increase amount was evidenced by the increase in mass. The determination of mole fraction of nitrogen absorbed was based on the assumption that mole of the specimen before annealing was equal to mole of the specimen after annealing, i.e.,

$$\frac{W_{before}}{M(Sm_nCo_m)} = \frac{W_{after}}{M(Sm_nCo_mN_x)} \qquad 4.1$$

where W_{before} and W_{after} were the weighed before and after annealing respectively, while $M(Sm_nCo_m)$ and $M(Sm_nCo_mN_x)$ were a molecular weight of the specimens respectively.

The crystal structure was determined with a Philips PW 1830/00 X-ray Diffractometer using the Cu K_α radiation. The X-ray data allowed us to determine the volume of unit cells and lattice constants. A least square fitting program and the reflective index associate with the angle of reflection were used to determine the lattice parameter.

The samples were pressed into a circular pellets of 9mm diameter and 4-5mm thickness in stainless steel compactor unit under 500 lb/in^2 uniaxial press, figure 4.3. This was done by mixing the ground powder with polyvinyl-alcohol 4-5% by weight in boiling water. The magnetic properties were measured with a Walker scientific magnetic Hysteresisgraph, figure 4.4. The saturation magnetization (obtained in Gauss unit) were converted to emu/g through the relationship

$$Magnetization(emu/g) = \frac{Magnetization(G)}{4\pi D} \tag{4.2}$$

where D was the density of the pellets. D was obtained by weight from the pellets in the air and water. The density of the pellets was obtained from

$$D = \frac{M_{air}\,\rho_{water}}{M_{air} - M_{water}} \tag{4.3}$$

By measuring M_s in unit of emu/g, we were able to find $\mu_B/F.U.$ or Bohr magneton per formula unit. This allowed us to determine the number of magnetic moment per formula through the relation

$$M(\mu_B / F.U.) = \frac{M_{molecular} \times 10^{21}}{9.27N} \times M(emu/g) \tag{4.4}$$

where N was Avogadro number, $M_{molecular}$ was the molecular weight of the specimen. The results of these experiment will be given in the next chapter.

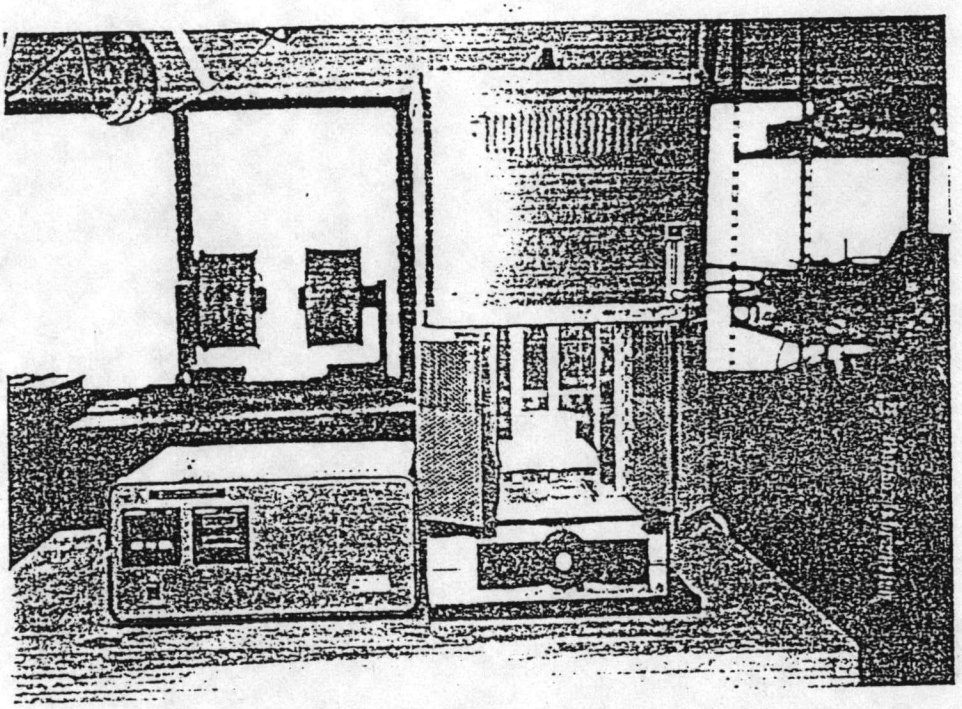

Figure 4.1 Linberg/Bluem crucible furnace model 567224.

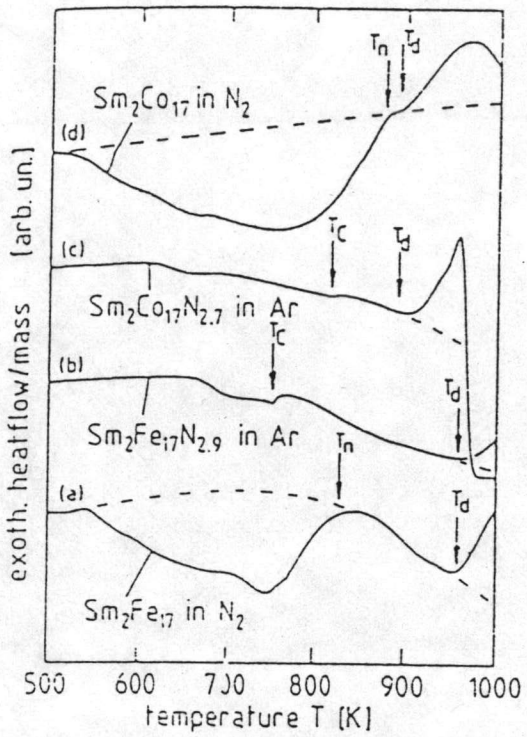

Figure 4.2 The nitrogen absorption rate of Sm_2Co_{17} compound and Sm_2Fe_{17} compound relates to annealed temperature which the absorption rate is maximum at the point of the DSC are minimum.

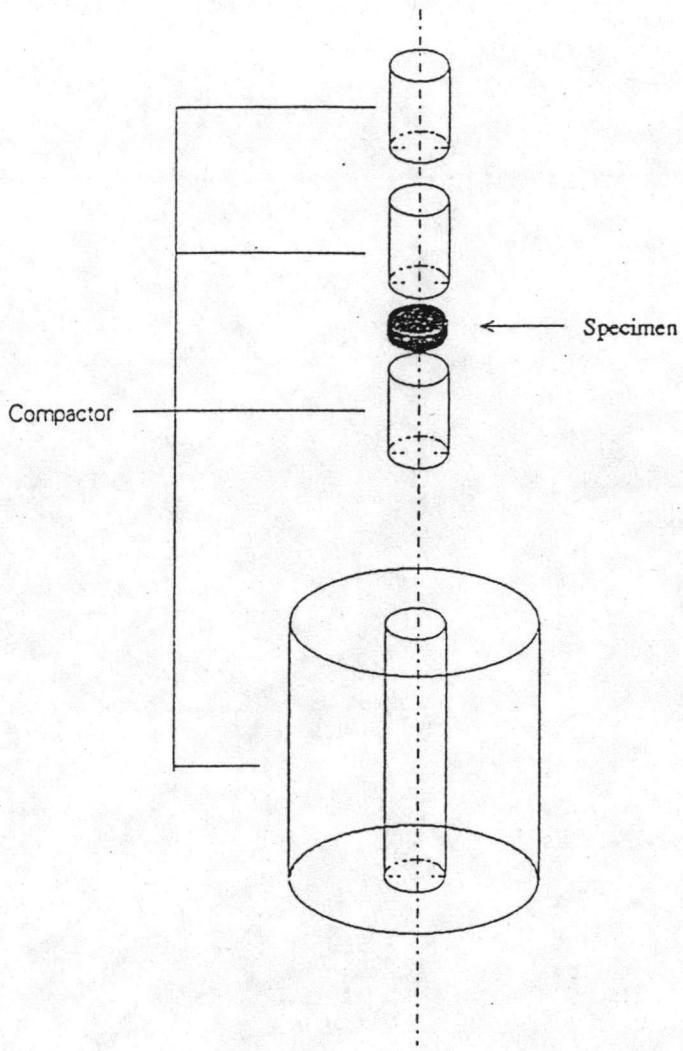

Figure 4.3 The compactor unit.

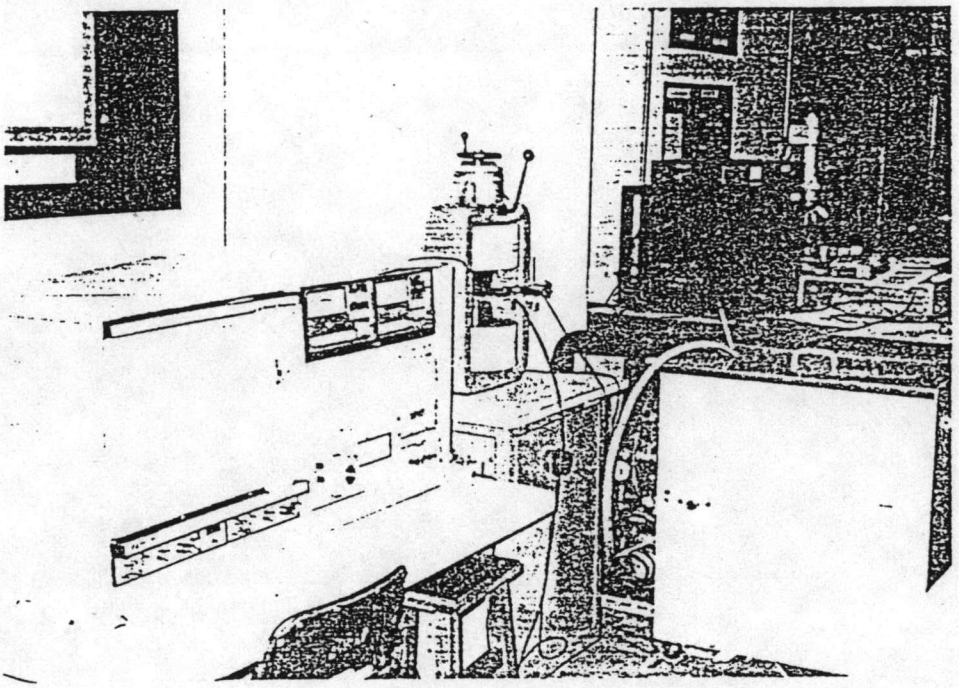

Figure 4.4 Walker Scientific Magnetic Hysteresisgraph.