V-6

Magnetic Properties and Phase Study of Mechanically Milled and Annealed $SmCo_5$ Alloy

(기계적 밀링 후 annealing한 $SmCo_5$ 합금의 자기적 특성 및 상관계)

D. H. Hwang*, Y. M. Chin, H. W. Kwon
황동하, 진영미, 권해웅

School of Materials Science and Engineering, Pukyong National University,
Busan, 608-739, Korea
부경대학교, 신소재공학부

1. Introduction

High-energy mechanical milling technique has been intensively studied as a useful means of producing magnetically coercive powder of rare earth – transition metal permanent magnetic alloys. In this technique, a pre-alloyed lump with desired composition is milled intensively and subsequently annealed to crystallize the hard magnetic phase with extremely fine grain structure. Through the mechanical milling work on a single-phase $SmCo_5$ alloy in our laboratory, we have been seen that the intensively milled amorphous-like $SmCo_5$ material has been crystallised not into the single 1:5 phase after crystallisation annealing, but rather, crystallised into a mixture of 2:17 and 1:5 phases. Even more interestingly, the phase relation in the annealed material has been found to be dependent upon the annealing condition. In this article, the phase relation and magnetic properties of a mechanically milled and annealed $SmCo_5$ material are investigated, and the thermomagnetic behaviour of a mechanically milled $SmCo_5$ alloy is also discussed.

2. Experimental

$SmCo_5$ alloy with composition 16.37 at% Sm and 83.63 at% Co was prepared by an induction melting, and the prepared alloy lumps were pulverized into a powder with grain size less than 100 μm. The pulverized alloy powder was charged into a hardened steel vial together with hardened steel balls. The mass ratio between the charged material powder and milling balls was 1:10. The charged vial was evacuated and then filled with a high purity argon gas. The material was then milled using a shaker mill. The milled powder was annealed in vacuum. Phase change in the material after the milling and subsequent annealing was examined mainly by an XRD (x-ray diffraction) using Cu Kα radiation. TMA (thermomagnetic analysis) and DTA (differential thermal analysis) was undertaken to investigate the crystallisation of the milled material. Magnetic properties of the annealed powder were measured by VSM (vibrating sample magnetometer). Prior to the VSM measurement, the sample was magnetised by applying pulsing field of 6 Tesla.

3. Results and discussion

The $SmCo_5$ alloy has been decomposed into an amorphous state after the mechanical milling for 36 hrs, and this amorphous material has been crystallized by the annealing at an elevated temperature. The annealed material has two-phase structure consisting of 1:5 and 2:17 phases rather than the initial 1:5 single-phase structure. The TMA tracing of the mechanically milled amorphous material showed that the magnetisation increases with increasing the temperature and then drops radically around 500 °C (Fig. 1). The magnetisation increase with increasing temperature at lower temperature range can be explained by the reduction of magnetic anisotropy of the amorphous material with increasing the temperature. The radical magnetisation drop at 500 °C is believed to be attributed to the crystallisation of amorphous phase with higher magnetisation into the crystalline Sm-Co compounds with lower magnetisation. The effect of annealing condition on the phase relation in the mechanically milled and annealed material was examined using an XRD, and a phase diagram showing the phase relation has been established based upon the findings (Fig. 2). It can be seen that the materials annealed above 500 °C has a crystallised from and two-phase structure. The major phase in the annealed two-phase material seems to be 2:17 type phase when it is annealed at lower temperature. The major phase changes to 1:5 type phase in the material annealed at higher temperature. Meanwhile, the material annealed below 400 °C still shows amorphous state.

The mechanically milled material after annealing at 600 °C shows very high coercivity, and this is due to the ultra finely crystallized Sm-Co compounds. The average grain size of this annealed material estimated from the XRD reflection broadening was around 12 nm. Meanwhile, the material annealed at 1000 °C has a radically reduced coercivity compared to that annealed at 600 °C, and this can be explained by an excessive grain growth. The materials annealed under different conditions shows different initial magnetisation curves. The material annealed at 600 °C for 20 min, which is thought to consist of a mixture of 2:17 and 1:5 type phases with 2:17 type phase being a major phase, shows the initial curve in which a likely coercivity mechanism is thought to be a pinning mechanism. In this material, the microstructure consists of ultra-fine grains (around 12 nm) and the domain wall (probably interaction domain wall) is thought to be pinned at the phase boundary between the two phases. Meanwhile, the material annealed at 1000 °C for prolonged time of 120 min, which consists of a mixture of 1:5 and 2:17 type phases with the 1:5 type phase being a major phase, shows the initial curve in which a likely coercivity mechanism is thought to be a nucleation mechanism. In this material the grains are believed to be over-grown, and a reverse domain may be nucleated more readily at the phase boundary between the two phases.

4. Conclusion

The mechanically milled amorphous $SmCo_5$ alloy was crystallised not into the single 1:5 phase after annealing, but rather, crystallised into a mixture of 2:17 and 1:5 phases. The phase relation in the annealed material was dependent upon the annealing condition. The materials annealed at lower temperature range above the crystallisation of the Sm-Co compounds had a two-phase structure consisting of 2:17 and 1:5 phases with the 2:17 type phase being a major phase. The material annealed at higher temperature also consisted of the two-phase structure. In this material, however, the major phase was 1:5 type phase. A likely coercivity mechanism in the mechanically milled and annealed material was dependent upon the annealing condition, and the governing coercivity mechanism in the materials annealed at lower and higher temperature range was found to be a pinning and nucleation-type, respectively.

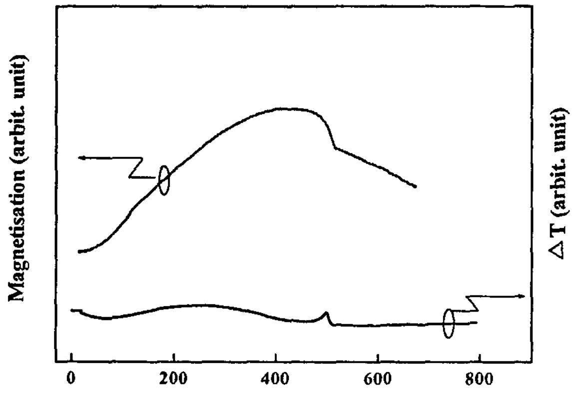

Fig. 1. TMA and DTA tracings of the $SmCo_5$ alloy mechanically milled for 36 hrs.

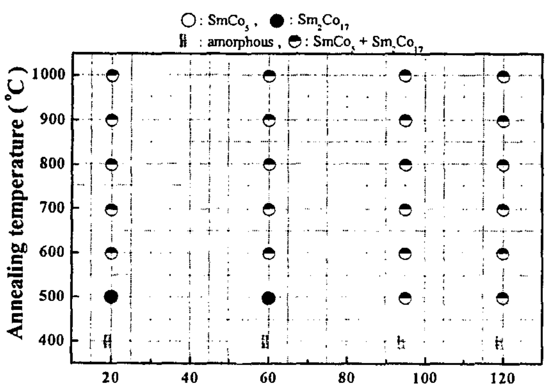

Fig. 2. Phase diagram of the $SmCo_5$ alloy mechanically milled (36 hrs) and then annealed at various conditions.