Coercivity and magnetic viscosity in mechanical milled nanocrystalline YCo_5

J. T. Elizalde Galindo, J. A. Matutes Aquino

Abstract

High coercivity and magnetic viscosity in nanocrystalline YCo5 alloy were observed. The alloy was obtained by arc-melting the raw materials, mechanical milling for 4 h with subsequent heat treatment for short times at 1103 K and a final quenching in water. The x-ray diffraction pattern showed a single hexagonal 1:5 phase. The average crystallite size determined by transmission electron microscopy was 13 nm. Magnetic measurements were carried out at 85 K in a pulsed field magnetometer varying the field in the range 50–250 kOe and keeping the pulse duration equal to 0.3 s. A maximum coercivity of 50 kOe was measured when applying a maximum magnetic field of 250 kOe. The viscosity parameter was measured varying the d*H*/d*t* rate in the range 511–1113 kOe/ s. The calculated activation volumes from viscosity data were smaller than the crystallite volume from transmission electron microscopy, consistent with their different physical meanings.

Introduction

Using pulsed magnetic field magnetometry it is possible to obtain the high magnetic fields necessary to study the magnetic properties of high anisotropy rare earth-transition metals alloys. The pulsed field technique can also be used to study magnetic viscosity phenomena in permanent magnet materials.[1] In particular with this technique magnetic viscosity phenomena have been studied in microcrystalline $Sm/Co_{1-x}Cux)_5$ and $Y(Co_{1-x}Cux)_5$ alloys.[1,2]

Recently we have obtained and studied the structural and magnetic properties of nanocrystalline YCo_5.[3] In this work we study the magnetic properties of nanocrystalline YCo_5 measured by pulsed field magnetometry at low temperatures.

Experiment

Raw material ingots with purity of 99.9% for Y (Alfa Aesar) and 99.8% for Co (Alfa Aesar) were used. Small buttons with a nominal composition of YCo_5 were produced by arc melting the raw materials under high purity Ar atmosphere. The buttons were melted four times to ensure homogeneity. The as-cast buttons were then coarsely pulverized and sieved with a 177 μm pore size sieve. The powders were mechanically milled during 4 h using a SPEX 8000 ball mill under argon atmosphere with a powder to ball ratio of 1:8. The as-milled amorphous powders were sealed in vycor ampoules under high vacuum and annealed at 1103 K for 2.5 min followed by quenching in water. An automated Siemens D5000 x-ray diffractometer with graphite monochromator and Cu *Kα* radiation was used to identify the present phases. Philips CM200 transmission electron mimicroscopy (TEM) was used for microstructural studies. A pulsed magnetic field magnetometer with a maximum field of 250 kOe was used to measure the magnetic properties at its operation temperature of 85 K.

Results and discussion

Figure 1 shows the x-ray diffraction pattern of YCo_5 after 4 h of mechanical milling and subsequent annealing for 2.5 min at 1103 K with water quenching. Peak broadening is observed due to a reduced crystallite size of YCo_5 phase. The asymmetry in the (110) and (002) peaks suggest a small quantity of $Y2Co_{17}$ secondary phase, but the peak indexing confirms that YCo_5 with hexagonal structure represents the

overwhelming majority phase in the annealed powders. The cell parameters, *a*=4.950(1) Å and *c*=3.973(1) Å, were determined from the x ray diffraction patterns by using the UNITCELL program.[4]

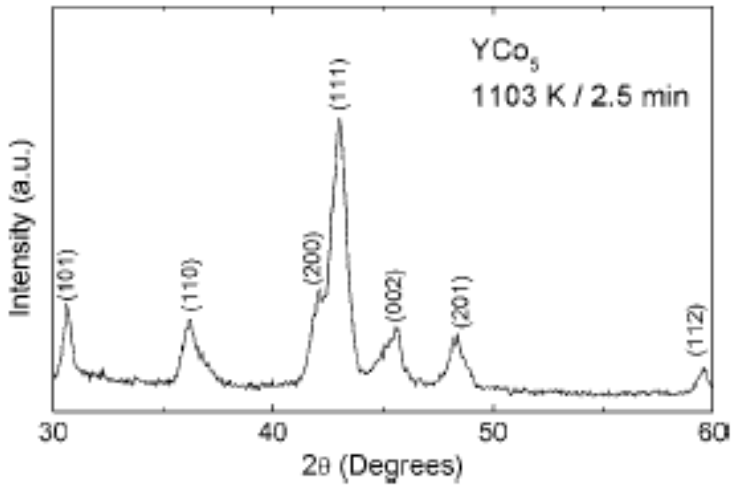

FIG. 1. X-ray diffraction pattern of YCo_5 after 4 h of mechanical milling and subsequent annealing for 2.5 min at 1103 K with water quenching.

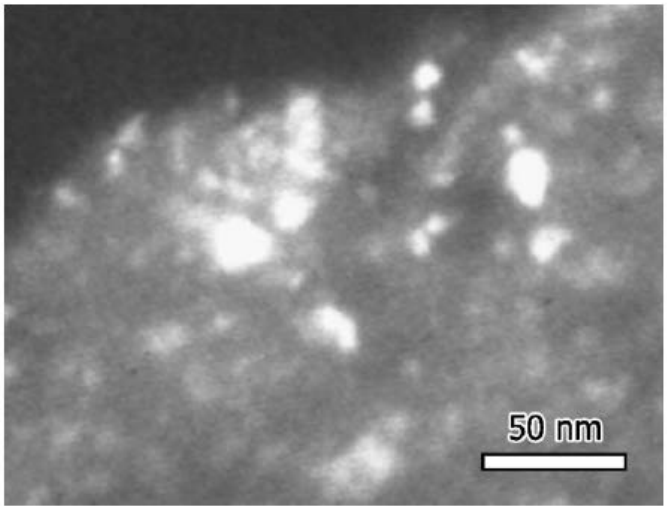

FIG. 2. Dark field TEM micrography of nanocrystalline YCo_5 mechanical milled by 4 h and annealed at 1103 K during 2.5 min.

Figure 2 shows a dark-field TEM micrograph of nanocrystalline YCo_5 mechanically milled for 4 h and annealed at 1103 K during 2.5 min. This micrography

shows the uniform microstructure of nanocrystalline YCo_5 within a powder particle. The average crystallite size of 13 nm determined by TEM in this work is in the appropriated range to obtain an important magnetic exchange coupling between adjacent crystallites, as it was shown in Sánchez Ll. et al.3 from the hysteresis loops, where an enhanced remanence with $\sigma r / \sigma_{max}$ bigger than 0.6 was observed.

On the other hand, the high coercivity values observed in Fig. 3 can be associated not only with the small crystallite size but also to the low thermal effects and to an increment of the anisotropy field at low temperatures near the liquid nitrogen temperature.[5] The increase of H_C with dH/dt, also shown in Fig. 3, is a consequence of strong magnetic viscosity effects in the nanometric-scale crystallites of YCo5 intermetallic compound. This result diverges from that given by Téllez-Blanco et al.[2] where the H_C field does not depend on the applied field rate dH/dt due to the micrometric-scale crystallites reported by them. The higher density of crystallite boundary in samples with nanometric-scale crystallites increases the magnetic viscosity effects compared with the lower density of crystallite boundary in samples with micrometric-scale crystallites.[6,7] These results confirm the important role of the microstructure in the observed magnetic properties of a given compound.

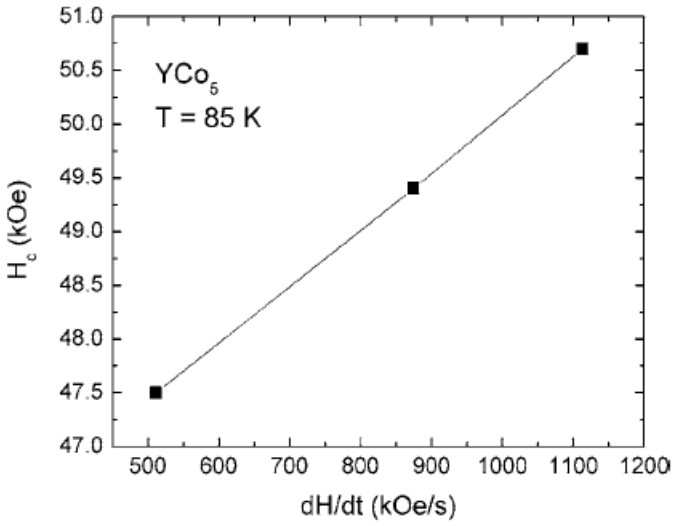

FIG. 3. Coercivity field values as a function of relative values of the applied field rate dH/dt.

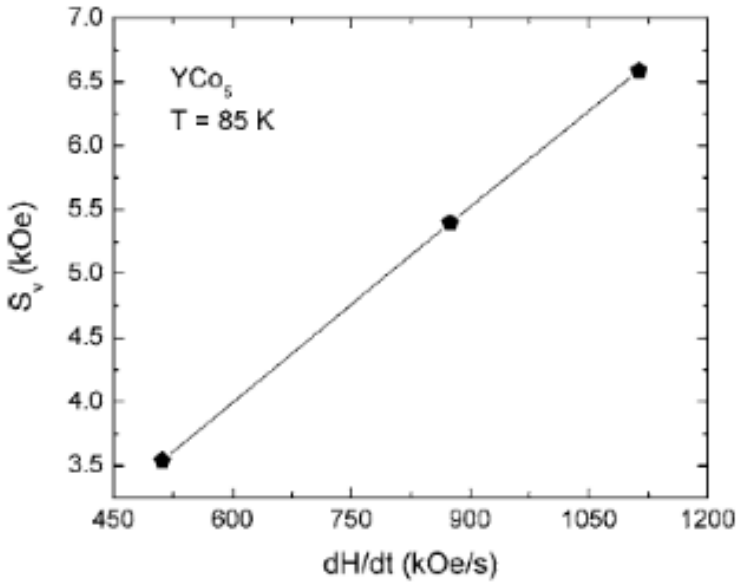

FIG. 4. Viscosity parameter S_v behavior with the increment of dH/dt. A linear increase of S_v with increasing dH/dt is shown.

The magnetic viscosity is characterized by the viscosity parameter $S_{v,}$ which is related to the coercivity H_C by the equation

$$S_v = \frac{\Delta H_C}{\ln(dH_{max,1}/dt)/(dH_{max,2}/dt)}, \quad (1)$$

where ΔHC is the increment of H_C for two maximum consecutive field values, $H_{max,1}$ and $H_{max,2}$, applied in two different pulses of equal duration (0.3 s), and dH/dt is the initial slope of the t-H curve for each of these two pulses.[1] Figure 4 shows the linear increase of the viscosity parameter S_v with d*H*/d*t* according to Eq. (1), where the highest value of S_v corresponds to the highest value of H_C.

The activation volume is related with S_v by the equation

$$v = \frac{kT}{\psi \mu_0 M_s S_v}. \quad (2)$$

Here Ψ is a geometrical factor of the order of unity, k is the Boltzmann constant, *T* is the measurement temperature, and $\mu 0 M_s$ is the saturation magnetization.[8] Figure 5 shows the activation volume values for the different values of d*H*/d*t*, as obtained by Eq. (2). The activation volume can be interpreted as the minimum material volume necessary to change the magnetization for each different pulse field rate, and this curve shows a reduction of the activation volume with the increment of pulse field rate.[9] In the sample there is a difference between the activation volumes shown in Fig. [5] and the crystallite volume as determined by TEM observations ($v_{crystallite}$=1.2X10^{-24} m^3), which reveals the conceptual difference between these two types of volumes. The activation volume is the volume that inverts its magnetization in a demagnetization process, which is not necessarily the same than the crystallite volume.[8,9]

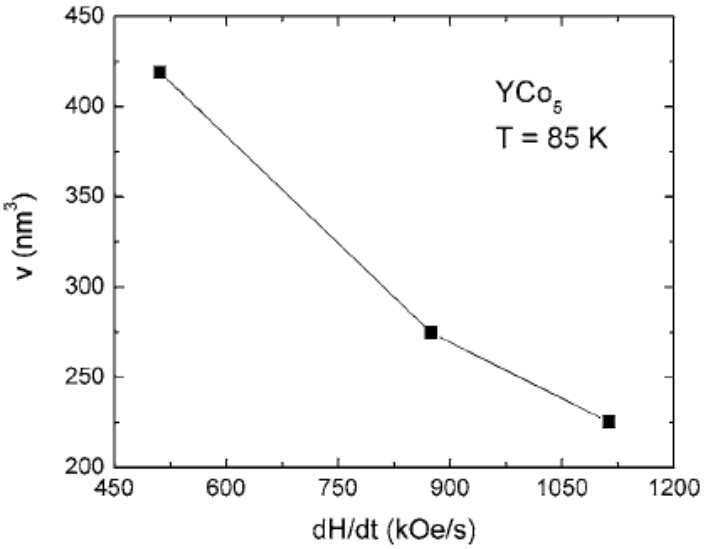

FIG. 5. Activation volume v values for the different values of dH/dt. A reduction of v with increasing dH/dt is observed.

Conclusions

It was shown that nanocrystalline YCo_5 powders with high coercivity and magnetic viscosity effect can be obtained by the mechanical milling technique. The largest value of coercivity of 50 kOe with a high $\sigma r / \sigma_{max}$ ratio >0.6 has been obtained for the YCo_5 powders milled for 4 h and annealed on high vacuum closed ampoules at 1103 K for 2.5 min and quenching in water. An average crystallite size around 13 nm was developed in the powders.

The high coercivity values observed in the YCo_5 powders at low temperatures near the liquid nitrogen temperature can be attributed not only to the small crystallite size produced by the mechanical milling and subsequent annealing, but also to the low thermal effects and to an increment of the anisotropy field.

The calculated activation volumes were smaller than the crystallite volume measured by TEM, in good agreement with the general acceptance that they have a different physical meaning**.**

Acknowledgments

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