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Orientation by solidification in a magnetic field A new process to texture SmCo compounds used as permanent magnets

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Abstract

The solidification of molten alloys in a static magnetic field is proposed as a new way of orienting polycrystalline materials. A high degree of orientation is obtained with samarium-cobalt compounds solidified in a static magnetic field. Whatever the cooling condition used from the liquid state, a magnetic field of several tesla induces crystallographic orientation in the solid. The easy magnetization axis of the polycrystal lies along the direction of the field applied during solidification. This texturing process is applied to the elaboration of Sm_2Co_{17} permanent magnets. Anisotropic bulk magnets with a coercive field up to 2250 kA/m and energy product above 160 kJ/m^3 are obtained. This process provides an alternative to the currently used industrial technology which is based on powder metallurgy. The paramagnetic susceptibility of the substituted Sm_2Co_{17} compounds is measured at high temperatures from which the susceptibility anisotropy at solidification temperature is determined. The orientation of the sample, solidified in a cold induction crucible, is analysed as a function of the applied magnetic field. Assuming a model in which particles are free to orient before complete solidification takes place, a critical size of these particles is deduced.

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Keywords: Permanent magnet; Anisotropy; Texture; Solidification in a magnetic field

1. Introduction

Rare earth permanent magnets are estimated to account for more than 30% of the sales value of the total world permanent magnet production. Among them, the SmCo magnets combine a high-energy product (BH_{max} ranging from 160–230 kJ/m^3) with very good corrosion and thermal stabilities. They have retained a strong industrial interest for applications which need magnets operating at elevated temperatures (up to 300°C) and in high demagnetizing fields (particle accelerators,

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high-frequency travelling wave tubes, servo-motors, application in aerospace, etc.).

Two hexagonal intermetallic compounds $SmCo_5$ and Sm_2Co_{17} are suitable for the production of permanent magnets. Indeed, these ferromagnetic phases combine the basic attributes of high saturation magnetization, high Curie temperature and large uniaxial magnetocrystalline anisotropy along the *c*-axis. Samarium-cobalt magnets can be divided into two kinds of materials: the single-phase magnets and the multi-phase (or substituted) magnets [1–3].

Single-phase $SmCo_5$ magnets have been used since the end of the 1960s. In these compounds, magnetization reversal occurs by nucleation and growth of reverse domains in individual grains. Coercivity is necessarily dependent on a very fine grained microstructure (grain size from 5–10 μm) [4–6], produced by powder metallurgical processing [1–3].

In multi-phase type SmCo magnets, based on $SmCo_5$ or Sm_2Co_{17} compounds, cobalt is partially substituted by copper. Consequently, the magnetization mechanism is radically changed: the coercivity is controlled by domain-wall pinning due to a second-phase precipitate induced by the copper. High coercive field can be achieved even in the as-cast state [4, 7]. As copper is a non-magnetic element, some iron is also substituted for cobalt in order to increase the magnetization of the alloy. The addition of a small quantity of zirconium also favours high coercivity [1–3].

The values of remanence and energy product are significantly enhanced when the magnets are anisotropic, i.e. when the crystallographic *c*-axis of the different elementary grains of the material are aligned. As conventional casting does not yield satisfactory orientation of the easy *c*-axis, directional solidification techniques have been tried [8–10]. They provided good orientation, but the process proved to be too complex and did not suit commercial production. Substituted SmCo magnets are still industrially produced by the powder metallurgical process.

Solidification in a magnetic field is a novel process which can produce oriented materials. Due to the persistance of magnetocrystalline anisotropy at high temperature, grains orient during solidification with their easy-magnetization axis along the direction of the applied magnetic field [11, 12]. Textured bulk samples of the high-temperature superconductor $YBa_2Cu_3O_7$ have been successfully produced in this way [13].

The purpose of this study is to show that oriented samples of samarium-cobalt compounds used for permanent magnets can be obtained by magnetic field processing. Even under unfavourable conditions of solidification (strongly uncontrolled thermal gradients, high cooling rate) high orientation is obtained. Bulk anisotropic substituted Sm_2Co_{17} magnets, with magnetic properties comparable to those of sintered magnets, are produced. This alternative process to the conventional powder-metallurgical technique could thus be a new way of making anisotropic permanent magnets. Furthermore, the good results obtained allow us to probe the microscopic nature of the orientation mechanism. In particular, we have succeeded in measuring the paramagnetic susceptibility anisotropy near the solidification temperature. Assuming a model in which solid particles are free to orient in the melt during the solidification process, the anisotropy energy of these particles is deduced from the field dependence of the sample orientation. This result is used to estimate a critical volume for the orienting particles.

2. Experimental apparatus and procedure

Precursor alloys are prepared by melting stoichiometric amounts of metal, corresponding to the alloy composition, in a cold-induction crucible. This operation takes place under a partial pressure of argon, in the absence of an applied static magnetic field. Ingots are cast into a metallic mould and do not exhibit any notable orientation.

Different compositions of the precursor alloys have been tested in this study (see Table 1). The crystal structures of these magnetic phases belong to the hexagonal system, and their *c*-axis constitute the easy magnetization direction due to a strong uniaxial magnetocrystalline anisotropy. The main magnetic properties of these compounds [14–18] are given in Table 1.

Solidification experiments in a magnetic field are made with samples obtained by melting these

Table 1
Atomic composition and intrinsic magnetic material parameters of several compounds used in this study: T_c is the ferromagnetic Curie temperature. M_s, H_A and k_1 are, respectively, the saturation magnetization, the anisotropy field and the effective anisotropy constant, given at room temperature

	Binary alloys		Substituted alloys		
	$SmCo_5$	Sm_2Co_{17}	$Sm(Co,Cu)_5$	$Sm(Co,Cu,Fe,Zr)_z$	
			A	B_1	B_2
Atomic composition	16.7% Sm 84.3% Co	11% Sm 89% Co	16.7% Sm 25% Cu 58.3% Co	10.7% Sm 7.1% Cu 53.6% Co 26.8% Fe 1.8% Zr	11.6% Sm 7% Cu 60.1% Co 19.4% Fe 1.8% Zr
M_s (Am^2/kg)	90	130	54	110–120	
$\mu_0 H_A$ (T)	52	5.4	40	5–10	
k_1 (kJ/m^3)	2×10^4	3×10^3	9×10^3	2.3×10^3–5×10^3	
T_c (°C)	710	917	510	800–850	

precursor alloys. A cold-crucible induction furnace (operating at 100 kHz) is inserted into the room temperature vertical bore (diameter 120 mm) of a superconducting coil cryostat. The coil provides, inside the bore, a vertical static magnetic field of up to 8 T. This system permits a rapid and clean melting of the alloys followed by solidification in a magnetic field H_t. The samples are solidified either directly in the cold crucible, or cast in a refractory mould. This process produces samples weighing between 20 and 60 g (with a volume density of about 8.4).

The grain sizes of the polycrystalline samples range between 50 and 1000 μm, depending on the cooling conditions. Chemical compositions are determined using SEM observations. We do not find any appreciable loss of samarium following preparation in the cold crucible, and we notice particularly a very low content of Sm_2O_3 or Zr(C,N) compared to what is usually observed in sintered samples [19, 20].

Grain orientation is magnetically characterized at room temperature on the whole sample by measuring its magnetization parallel and perpendicular to the direction of the texturation field H_t. These measurements are carried out in a fluxmeter (in an open flux circuit) and give a volumetric estimate of the orientation.

The samples in this as-cast state do not exhibit a sufficient coercivity, though solidified in a magnetic field. They have to be magnetically hardened by being subjected to appropriate annealing [2, 3, 15, 20]. These treatments are carried out in an electrical furnace, under a secondary vacuum, in zero magnetic field. The samples are wrapped in stainless-steel foil. The high-temperature treatments do not affect the orientation of the samples.

3. Production of oriented samples

Solidification in the presence of a magnetic field of several tesla induces orientation of SmCo materials as is shown by the examples given below. The easy magnetization axis of the samples is parallel to the direction of H_t, which means that the c-axes are preferentially aligned along this direction.

The first example of this texturation process (sample S_1) is that of a $SmCo_5$ precursor alloy melted in a hemispherical cold crucible and cooled, by turning off the power of the furnace, in a magnetic field of 2.5 T. Since the copper crucible was cooled by water circulation, solidification occurred within only a few seconds. However, this quench does not prevent the orientation by the magnetic

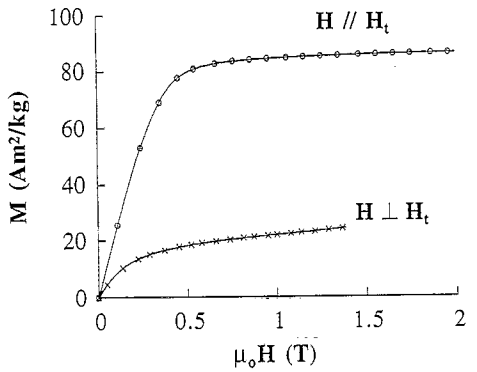

Fig. 1. Magnetization curves at room temperature for two perpendicular directions of the sample S_1 (m = 30 g of binary alloy $SmCo_5$). The sample is anisotropic and oriented with its easy-magnetization direction parallel to the field H_t applied during the solidification.

field as is shown by magnetic measurements (Fig. 1) and by X-ray diffraction pole figure (Fig. 2a and Fig. 2b). The strong correlation between these two characterizations prove the crystallographic orientation of the sample.

In the second example, the samples S_2 and S_3 were melted from a precursor alloy of composition B_1 (see Table 1), then cast in an alumina cylindrical mould (diameter 10 mm, length 15 mm). Sample S_2 was cooled in zero magnetic field, while S_3 was cooled in a field of 5 T. The magnetic measurements clearly show (see Fig. 3) the strong effect of the magnetic field applied during casting. Thus, thanks to the magnetic field, it should be possible to produce bulk oriented samples with different shapes, simply by casting in an appropriate mould.

In the third example, 20 different samples with substituted '2 : 17' composition $Sm(Co,Fe,Cu,Zr)_z$ were melted in a cylindrical cold-induction crucible and cooled in a magnetic field of 5 T. The bulk cylindrical ingots (diameter 20 mm, weight 30 to 60 g each) are oriented with the easy magnetization axis directed along their axial length. We do not observe any notable influence of the composition on the texturation. The orientation parameter τ is the ratio M_r/M_s where M_s is the saturation magnetization of the compound, and M_r is the remanent magnetization in the direction of H_t (when the

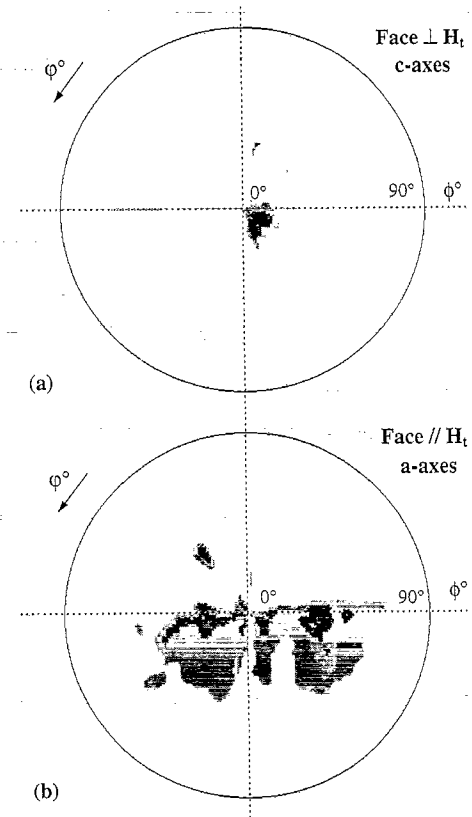

Fig. 2. X-ray diffraction pole figures measured on two faces of the sample S_1, cut perpendicular and parallel to H_t. The X-ray texture analyses were performed on a four-circle diffractometer in the Schulz reflection geometry. Pole figures were measured by scanning the tilt angle ϕ between 0 and 72°, and the azimuthal angle φ between 0 and 360°. The figures are the projection of the diffraction intensity of a given Bragg reflection system as the sample is rotated. The c-axis pole figure (a) reveals at least two crystallographic domains of close orientation, deviated by no more than 10° from H_t, as a strong evidence for the magnetic field effect. The a-axis pole figure (b) confirms the c-axis orientation as the poles are aligned on the horizontal main axis of the figure.

sample is not coercive, this remanent value is given by extrapolating the slope of the demagnetization curve). Due to the strong magnetocrystalline anisotropy of the compounds, τ reflects the degree of grain alignment. τ tends to 1 for a perfect alignment (i.e. if all the c-axis are oriented parallel to the direction of H_t) and to 0.5 if the grains form a random distribution [21, 22]. Fig. 4 is a plot of the

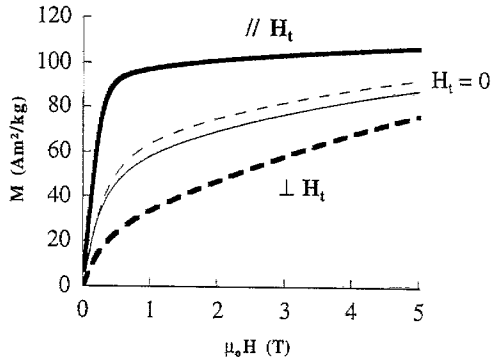

Fig. 3. Magnetization curves at room temperature, in two perpendicular directions, characterizing the volume orientation of the samples S_2 and S_3 ($m = 25$ g each) cast in a cylindrical mould. Sample S_2, cooled in zero magnetic field, does not exhibit any significant orientation (fine line). Sample S_3, solidified in 5 T, is oriented with its easy magnetization direction parallel to H_t (bold line).

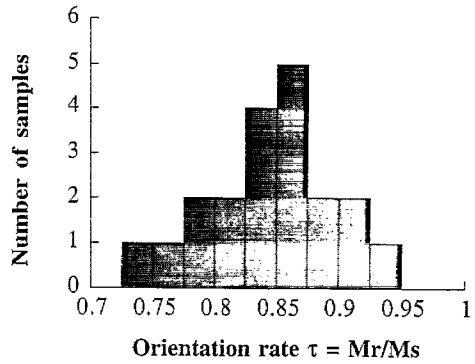

Fig. 4. The orientation distribution of 20 different samples made with substituted alloys $Sm(Co_xFe_{1-x}Cu_{0.08}Zr_{0.02})_z$, x ranging from 0.7–0.78 and z from 7.6–8.35. All the samples were melted in a cylindrical cold induction crucible (diameter 20 mm) and solidified in 5 T. For an isotropic sample $\tau = 0.5$, while $\tau = 1$ for a perfectly oriented sample. The results show that the great majority of the samples are very-well oriented ($\tau > 0.8$).

values of τ for these 20 samples. The results show the high success of the process: more than 80% of the samples are very well-oriented with an orientation factor τ greater than 0.8. The average value of τ is greater than 0.85. This corresponds to a magnetization loss

$$\frac{M_s - M_r}{M_s} < 15\%,$$

which is a very good value for a bulk magnet. The relatively low orientation ($\tau < 0.8$) of a few samples can be explained by their incomplete melting due to the experimental conditions.

Magnetization measurements have been carried out on pieces cut from different parts of a sample which has $\tau = 0.85$. The inner part of the sample has a significantly higher orientation than the outer part ($\tau_{inner} > 0.9$, $\tau_{outer} < 0.7$). This texturation distribution can be explained by the melting conditions in the cylindrical cold-induction crucible. The alloy experiences magnetic forces due to the radial gradient of the static magnetic field. These forces tend to push the material against the cold walls of the crucible, which gives rise to incomplete melting in this region and therefore to an incomplete orientation. Hence, the orientation measured on the whole sample, which is an average, is decreased by

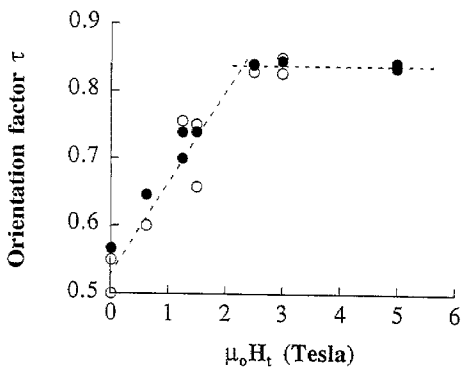

Fig. 5. Orientation factor $\tau = M_r/M_s$ as a function of H_t, for samples melted and cooled in a cylindrical cold induction crucible, in a vertical magnetic field H_t. The black and the white points represent samples with compositions B_1 and B_2, respectively. The evolution of the orientation is the same for the two compositions.

these edge effects. However, these negative effects can be reduced by using another shape of cold crucible (for example, a hemispherical crucible) or by using larger volumes of material.

The orientation of the bulk cylindrical samples (with compositions B_1 and B_2), made in the cylindrical cold-induction crucible, is plotted as a function of the field applied during solidification (see Fig. 5). The samples solidified without static

magnetic field do not exhibit any appreciable orientation (τ is about 0.5). The orientation factor τ increases with H_t and reaches a saturation value of 0.85 when H_t is equal or greater than 2.5 T. The theoretical maximum saturation value $\tau = 1$ is not attained because of the edge effects which the magnetic field cannot overcome in this experiment setup.

4. Bulk anisotropic permanent magnets

The demagnetization curves of two well-oriented bulk samples, magnetically hardened by specific annealings, are plotted in the Fig. 6a and Fig. 6b. The good squareness of the hysteresis loop of the permanent magnet made with the composition A (see Fig. 6a) leads to an energy product $BH_{max} = 57\ kJ/m^3$. This value is close to the maximum theoretical value $B_r^2/4\mu_0 = 58\ kJ/m^3$. The relatively low value of the remanence $\mu_0 M_r = 0.55$ T, measured despite a very good orientation ($\tau = 0.95$), is due to the high quantity of copper in the alloy (Table 1). However, the magnetic properties of this bulk anisotropic magnet are quite equivalent to those of the sintered magnet made with the same atomic composition.

The permanent magnet made with the compound B_1 (see Fig. 6b) combines high remanence magnetization ($\mu_0 M_r = 1$ T, thanks to a good orientation $\tau = 0.88$) and very high coercivity ($\mu_0 H_c = 2.8$ T). These properties lead to an energy product BH_{max} greater than $160\ kJ/m^3$.

These two results show that it is possible to obtain anisotropic bulk permanent magnets with high-quality magnetic properties. These bulk magnets can be made as energetic as sintered magnets provided suitably adapted compositions of precursor alloys are used. This is necessary because the bulk process described in this paper leads to lower loss of samarium and zirconium than that which occur during sintering. So different precursor compositions are required to achieve the same result.

5. Attempt to explain the orientation mechanism

A magnetically anisotropic material in a magnetic field tries to minimize its anisotropy energy

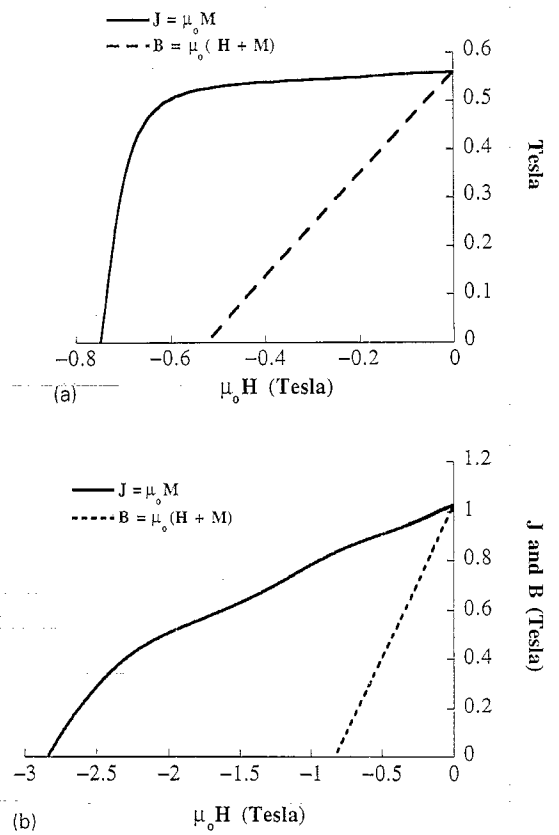

Fig. 6. Demagnetization curves of two bulk permanent magnets made with the composition A (a) and the composition B_1 (b). The anisotropic samples ($m = 25$ and 35 g, respectively) were solid-solution heat treated for 5 h at 1150°C. After a quench to room temperature, the samples were aged at 850°C for 20 h (800°C for 10 h, respectively), followed by a slow cooling to 400°C at a rate of about 100°C/h (about 10°C/h, respectively), then quenched again.

E_θ. In the case of a paramagnetic or a diamagnetic crystal, E_θ can be written

$$E_\theta = -E\cos^2\theta = -Ve\cos^2\theta$$

V is the volume of the particle and e is its volume anisotropy energy

$$e = \tfrac{1}{2}\mu_0\Delta\chi_V H_t^2.$$

$\Delta\chi_V$ is the difference between the magnetic susceptibilities in two crystallographic directions (magnetocrystalline anisotropy) and θ is the angle between the field H_t and the direction of the

greatest susceptibility [11]. The crystal is subjected to a magnetic torque Γ:

$$\Gamma = -\frac{dE_\theta}{d\theta} = -E \sin 2\theta.$$

If the crystal can move freely, its direction of largest susceptibility will tend to become parallel to the direction of the magnetic field.

At the beginning of the solidification, it is assumed that some free nuclei are crystallizing in the melt. If the anisotropy of these particles is strong enough, the applied field will induce an orientation of the crystal growth.

The paramagnetic susceptibility per unit mass of a '2 : 17' substituted compound has been measured as a function of the temperature (see Fig. 7), using a high-temperature susceptometer developed in the laboratory [23]. The starting sample is a part of the initial precursor alloy of composition B_1 which is isotropic. The mass susceptibility χ measured while increasing the temperature corresponds to an average of the different values of χ along the different crystallographic directions of the material:

when $T = 1175°C$, $\chi_{iso} = 5.45 \times 10^{-7}$ m^3/kg.

When the temperature is high enough, the sample melts:

when $T = 1250°C$, $\chi_{liquid} = 4.2 \times 10^{-7}$ m^3/kg.

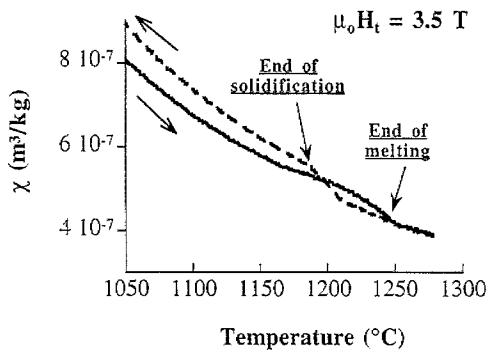

Fig. 7. Paramagnetic mass susceptibility χ as a function of the temperature for a sample of composition B_1, melted and solidified in a magnetic field $\mu_0 H_t = 3.5$ T. The value of the susceptibility increases before and after the solidification which characterizes the orientation of the sample. It gives an estimate of the susceptibility anisotropy of the compound near the solidification temperature: $\Delta\chi = 3 \times 10^{-8}$ m^3/kg.

When the temperature is decreasing, the compound solidifies. As the solidification occurs in a magnetic field of 3.5 T, the sample is oriented: at the same temperature, the measured susceptibility is higher than what it was before melting because it corresponds to the c-axis value of χ:

when $T = 1175°C$, $\chi \| c = 5.75 \times 10^{-7}$ m^3/kg.

The change in the susceptibility gives the value of the anisotropy of the paramagnetic susceptibility near the temperature of solidification:

$$\Delta\chi = 3 \times 10^{-8} \text{ m}^3/\text{kg}.$$

With a density value $d = 8.4$ (the room temperature value for these compounds):

$$\Delta\chi_V = 2.5 \times 10^{-4}.$$

According to the value of $\Delta\chi_V$, the volume anisotropy energy of a solid particle present in the melt during the solidification process is equal to 620 J/m^3 or to 2490 J/m^3 if H_t is equal to 2.5 or 5 T, respectively. If the energy E is large enough (that is if the values of e or V are large enough), the nucleus will put its c-axis parallel to H_t, making oriented polycrystalline samples.

To simplify, we suppose that the crystallizing nuclei are free in the melt, and we neglect disturbing factors such as viscous forces, convective flows in the melt and interactions between nuclei and between the melt and the walls of the crucible (edge effects). In this simple model, the orientation mechanism is a competition between the anisotropy energy and the thermal activation kT, where k is the Bolztmann constant ($k = 1.38 \times 10^{-23}$ J/K) and T is the solidification temperature. If $T = 1200°C$, E has to be greater than $kT = 2 \times 10^{-20}$ J.

The average of the normalized projections of the individual-grain c-axis along the measurement direction (which is here the direction of H_t) is $\langle \cos\theta \rangle$. When the sample solidifies in the magnetic field, the c-axes of the different grains take an angular distribution represented by $\langle \cos\theta \rangle$. This distribution remains the same at room temperature and $\langle \cos\theta \rangle$ corresponds to the orientation rate $\tau = M_r/M_s$.

Assuming an axisymmetric distribution of the c-axis, with respect to the direction of H_t, $\langle \cos\theta \rangle$

can be calculated using the Boltzmann distribution $f(\theta) = e^{-E_\theta/kT}$:

$$\langle \cos\theta \rangle = \frac{\int_0^{\pi/2} \sin\theta \cos\theta \, e^{a(\mu_0 H_t)^2 \cos^2\theta} \, d\theta}{\int_0^{\pi/2} \sin\theta \, e^{a(\mu_0 H_t)^2 \cos^2\theta} \, d\theta}.$$

$\tau = \langle \cos\theta \rangle$ can be written as a parametric function of $(\mu_0 H_t)^2$; $\tau = F_a(\mu_0 H_t^2)$ where the parameter a is $a = \Delta\chi_V V/2\mu_0 kT$.

In accordance with the free nuclei approximation, the experimental values of τ plotted in Fig. 5 can be corrected for the edge effects and normalized to the saturation value $\tau = 1$. These values are plotted as a function of $(\mu_0 H_t)^2$ (see Fig. 8). The curve is very well fitted with the numerically calculated function F_a, with $a = 3\ T^{-2}$. Choosing an average solidification temperature $T = 1200°C$, the result permits the evaluation of the orientation volume V_o of the free particles in the melt:

$V_o = 7 \times 10^{-4}\ \mu m^3$.

The discussion developed here is based on the assumption that each grain of an oriented bulk sample has grown from a nucleus which was present in the melt and has been oriented by the magnetic field during the solidification process. These nuclei have to be free to orient their c-axis along the field, which supposes that the solidification is governed by a homogeneous nucleation process: the nuclei with the critical nucleation size (below which a nucleus is dissolved in the melt) are growing inside the crystallization interval which exists for these SmCo compounds [24, 25]. The orientation volume V_o, evaluated experimentally, is then necessarily greater than the critical nucleation volume.

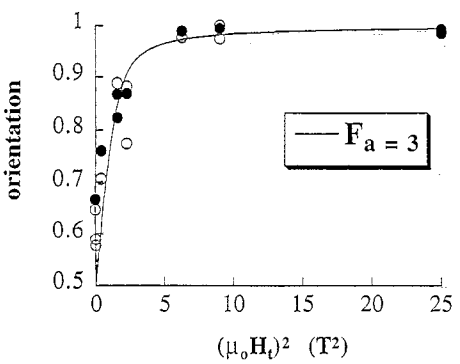

Fig. 8. The average $\langle \cos\theta \rangle$ is calculated with a Boltzmann distribution $e^{-E_\theta/kT}$. The experimental values of the orientation factor τ (corrected for edge effects) of the samples solidified in a field H_t are very well fitted by the function F_a with $a = 3\ T^{-2}$.

The magnetocrystalline anisotropy energy of the particles with such a volume V_o is equal to $E = 18kT = 3.6 \times 10^{-19}$ J, if the applied magnetic field is $\mu_0 H_t = 2.5$ T. This energy is strong enough to induce the particles orientation. A critical volume V_c, corresponding to this energy value, can be defined for each value of the applied magnetic field. Then particles with size lower than this critical size V_c do not have enough anisotropy energy to orient. In the case of our study (Fig. 5), the estimated volume V_o corresponds to the value of V_c for 2.5 T.

During their growth, the nuclei necessarily reach the size which should give them enough anisotropy energy to orient in a magnetic field lower than 2.5 T. Indeed, applying, for example, a magnetic field 10 times smaller ($\mu_0 H_t = 0.25$ T), the volume V_c is only equal to 0.07 μm^3; this value is about 10^7 times smaller than the typical grain size of a solidified sample. However, samples solidified in a magnetic field below 2 T are not well-oriented (Fig. 5). In such fields, the orientation volume V_c being larger than V_o, the nuclei reach their orientation size when the solidification process is almost over. Consequently, the particles are no longer sufficiently free to orient (because they have not enough time to turn before complete solidification occurs or they do not have enough space, a fraction of the alloy being already solidified). The orientation mechanism is then effective only if it occurs at the beginning of the solidification process, which is consistent with the small value of V_o.

Another interpretation could be the existence of residual metastable solid particles in the liquid a few degrees above the melting temperature [12]. These particles would be anisotropic enough to orient in a magnetic field (with a volume V_o) and would act as nuclei for oriented solidification of the crystal. In this way, the orientation would occur before the solidification process. This could explain why orientation by solidification in a magnetic field is obtained whatever the cooling conditions are.

6. Conclusion

Successful results have been obtained with the binary compounds $SmCo_5$ and Sm_2Co_{17}, and also with the substituted compounds $Sm(Co,Cu)_5$ and $Sm(Co,Cu,Fe,Zr)_z$. Oriented samples are obtained, by solidification in several tesla, even in unfavourable conditions of solidification thanks to a strong anisotropy of the paramagnetic susceptibility of the compounds: $\Delta\chi = 3 \times 10^{-8}$ m^3/kg measured at $T = 1175$°C with substituted '2 : 17' alloys. Assuming a model of solid particles existing in the melt, a critical size for the orientation of these particles in a magnetic field is estimated: V_o is about 10^{-3} μm^3 in the case of solidification in a cold-induction crucible. Consistent with this small volume, the particles may be the primary nuclei crystallizing at the beginning of the solidification process, which are sufficiently anisotropic and free to orient in a magnetic field, in the melt before complete solidification occurs. The physical interpretation of the orientation mechanism apart, the high success rate of the process may lead to industrial applications. Indeed, solidification in a magnetic field, followed by appropriate annealings, has proven to be a promising new process for the manufacture of bulk anisotropic permanent magnets.

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