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<p>The effect of ball milling in the microstructure and magnetic properties of Pr₂Fe₁₇ compound</p> <p>The effect of a severe mechanical milling treatment on the microstructure, magnetic and magneto-caloric properties of Pr₂Fe₁₇ powders is reported. Bulk alloys showing a rhombohedral Th₂Zn₁₇-type crystal structure were mechanically ball milled under Ar atmosphere. After 10 h of milling this crystal structure persists and the mean values of the lattice parameters remain almost unchanged. Average grain sizes around 27 nm were estimated by both, transmission electron microscopy and neutron powder diffraction measurements. While for the starting bulk alloys the low field temperature of the magnetization, M(T), shows a well- defined and sharp decrease at the Curie temperature, TC = 285(2) K, in ball milled samples the transition becomes broad not allowing an accurate determination of Curie point; in addition, this intrinsic parameter seems to be shifted toward a higher</p>	<p>Ảnh hưởng của quá trình nghiền bi đến tính chất vi cấu trúc và tính chất từ của hợp chất Pr₂Fe₁₇</p> <p>Chúng tôi nghiên cứu ảnh hưởng của quá trình nghiền cơ học mãnh liệt đến các tính chất vi cấu trúc, tích chất từ và từ nhiệt của bột Pr₂Fe₁₇. Các kim loại dạng khối có cấu trúc tinh thể dạng hình thoi Th₂Zn₁₇- được nghiền bi cơ học trong môi trường khí Ar. Sau 10 giờ nghiền, cấu trúc tinh thể này vẫn tồn tại và các giá trị trung bình của các tham số mạng hầu như không đổi. Thực hiện cả phép đo kính hiển vi điện tử truyền qua và nhiễu xạ bột nơ tron, chúng tôi thấy kích thước hạt trung bình khoảng 27 nm.</p> <p>Trong khi đối với các hợp kim dạng khối ban đầu, nhiệt độ từ hóa trường thấp M(T) thể hiện sự giảm rõ nét ở nhiệt độ Curie TC = 285(2) K, trong các mẫu được nghiền bi, sự dịch chuyển rộng hơn không cho phép xác định chính xác điểm Curie;</p> <p>Thêm vào đó, dường như tham số nội tại này dịch chuyển về</p>	
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temperature [292(10) K]. The magnetocaloric effect at $\Delta H_{max} = 5T$ was evaluated from the temperature dependence of magnetic entropy change, through the variation of $M(H,T)$ curves. A decrease in the peak value of magnetic entropy change, ΔS^{TM} , from 5.7 to 3.7 J kg⁻¹ K⁻¹, and the broadening of the maximum is observed for the milled sample respect to the bulk alloy.

Pr₂Fe₁₇ is a ferromagnetic compound that crystallizes in the rhombohedral Th₂Zn₁₇-type crystal structure (R3m), exhibiting high values of the spontaneous magnetisation and a Curie point, TC, of 283 K [1]. Recently, this material has attracted a renewed interest because combines a significant magneto-caloric effect close to room temperature with low potential production cost due to its high Fe content. A magnetic entropy change value, ΔS^M , around 6Jkg⁻¹ K⁻¹ at $J_u, \Delta H_{max} = 5T$ has been reported, while the related adiabatic temperature change, ΔT_{ad} , was roughly estimated in 4.1 K [2-4].

phía nhiệt độ cao [292(10) K]. Chúng tôi ước tính hiệu ứng từ nhiệt ở $\Delta H_{max} = 5T$ qua sự phụ thuộc nhiệt độ của độ biến thiên entropy từ, thông qua các biến thể của đường cong $M(H, T)$. Sự suy giảm giá trị peak của độ biến thiên entropy từ, ΔS^{TM} , từ 5.7 đến 3.7 J kg⁻¹ K⁻¹, và sự mở rộng cực đại xuất hiện ở các mẫu được nghiền so với các hợp kim dạng khối.

Pr₂Fe₁₇ là một hợp chất sắt từ kết tinh ở dạng cấu trúc tinh thể mặt thoi Th₂Zn₁₇ (R3m), có giá trị từ hóa tức thời và điểm Curie TC cao, 283 K [1]. Gần đây, vật liệu này đã được quan tâm trở lại vì nó có nhiều ưu điểm như hiệu ứng từ nhiệt mạnh gần nhiệt độ phòng cũng như giá thành sản xuất thấp do hàm lượng sắt cao của nó. Chúng tôi đã xác định được độ biến thiên entropy từ, ΔS^M , quanh 6Jkg⁻¹ K⁻¹ tại $J_u, \Delta H_{max} = 5T$, trong khi sự thay đổi nhiệt độ đoạn nhiệt tương ứng, ΔT_{ad} , theo ước tính khoảng 4,1 K [2-4].

Ball milling has been widely used as a technique for producing nanostructured or new metastable phases from pure elements [5] or bulk stable compounds, exhibiting a rich variety of novel physical properties compared with those of bulk starting material [6-8].

In this report we describe the effect of a severe ball milling treatment on structural, magnetic and magnetocaloric properties of Pr₂Fe₁₇ powders. Nearly single-phase Pr₂Fe₁₇ alloys were processed by high-energy ball milling during 10 h. A comparative study, by means of neutron powder diffraction (NPD), scanning (SEM) and transmission (TEM) electron microscopy, and magnetization vs. temperature and applied magnetic field measurements, of both, the starting and ball milled (BM) alloy is presented.

As-cast pellets of nominal composition Pr₂Fe₁₇ were prepared by Ar arc melting from 99.9% pure Pr and 99.98% pure Fe. To produce a highly pure 2:17 phase alloys were wrapped in tantalum foil, sealed under vacuum in quartz ampoules,

Nghiên bi đã được sử dụng rộng rãi như một kỹ thuật để tạo pha cấu trúc nano và siêu bền mới từ các nguyên tố tinh khiết [5] hoặc các hợp chất ổn định dạng khối, thể hiện sự đa dạng về tính chất vật lý so với các vật liệu khối ban đầu [6-8].

Trong báo cáo này, chúng tôi mô tả ảnh hưởng của phương pháp xử lý nghiền bi mãnh liệt đến đặc tính cấu trúc, từ và từ nhiệt của bột Pr₂Fe₁₇. Các hợp kim Pr₂Fe₁₇ gần một pha được xử lý bằng phương pháp nghiền bi năng lượng cao trong 10 giờ. Chúng tôi trình bày một nghiên cứu so sánh thông qua các phép đo nhiễu xạ bột neutron (NPD), kính hiển vi điện tử quét (SEM) và kính hiển vi điện tử truyền qua (TEM), và độ từ hóa theo nhiệt độ và từ trường tác động vào của cả hợp kim ban đầu và hợp kim nghiền bi (BM).

and homogenised during one week at 1373 K; the thermal treatment was followed of water quenching. Annealed samples were broken into smaller pieces and manually pulverised using an agate mortar. The obtained powder was sieved using a 106 μm pore size metallic sieve to be sealed in a stainless steel vial under argon atmosphere. A ball-to-powder weight ratio of 8:1 was chosen. Powder was dry milled during 10 h using a high energy Retsch PM/400 planetary ball mill. The process was carried out in successive steps of 5 min of milling followed of 5 min of break, in order to keep low the temperature to favour progressive grain size diminution.

Room temperature NPD patterns were collected on the D1B two- axis neutron diffractometer (ILL, Grenoble, France) using a neutron wavelength of $k = 2.52 \text{ \AA}$. The Rietveld analysis of the diffraction patterns has been performed using the Fullprof package [9], in order to make a quantitative determination of structural parameters and phase composition. Powder

morphology was characterised with Fig. 1. Experimental (dots) and calculated (solid line) neutron powder diffraction patterns of Pr₂Fe₁₇ alloys: (a) starting bulk alloy, (b) after 10 h of milling. Positions of the Bragg reflections are represented by vertical bars (the first vertical row corresponds to the crystal structure of Pr₂Fe₁₇ (see text), while the second one is associated with a-Fe). The observed-calculated difference is depicted at the bottom of the patterns.

aJeol modelJSM-6100 scanning electron microscope (SEM), while the microstructure of milled particles was investigated by means of aJeol 2000 EXII high resolution transmission electron microscope (TEM).

Magnetization measurements were performed in the temperature interval of 5-350 K, using a Quantum Design PPMS-14T platform with the vibrating sample (VSM) magnetometer module. For the characterisation of bulk alloy a bar-shaped sample of around 1 mm x 1 mm x 5

mm, was prepared. The low field $M(T)$ curves were recorded at $\mu_0 H_{ext} = 5$ mT with a temperature heating rate of 2K/min. The measurements were done on thermally demagnetized samples. Curie points were inferred from the minimum in the dM/dT vs. T curves. For the determination of the magnetic entropy change, $|\Delta S_M|$, a set of $M(H)$ curves was measured from 0 to 5 T in 0.1 T steps from 260 to 340 K. The magnetic entropy change was calculated by using the well-known relation:

Fig. 1 compares the NPD patterns of homogenized bulk and as-milled (10 h-BM) Pr_2Fe_{17} samples. The patterns have been refined considering two crystalline phases: the first one is a Th_2Zn_{17} -type rhombohedral crystal structure associated with the Pr_2Fe_{17} phase and the second one is related to a small amount of α -Fe impurity phase. The diffraction pattern for the starting bulk alloy is characterized by high intensity and sharp reflections. As shown in Fig. 1(b), milling leads to the significant broadening,

overlapping, and reduction in the intensity of diffraction peaks; in addition, a perceptible increase in the background baseline of the diffraction pattern occurs. These effects reflect the disordering introduced during the milling process, which is usually expressed as vacancies, dislocations, grain boundaries and chemical disorder [6,10].

In Table 1 we report the mean cell parameters deduced from the profile refinement of whole diffraction pattern, accompanied by a summary of magnetic data. The values obtained are in perfect agreement with those reported in [1,11]. The R-factors obtained for the analysis reflects its satisfactory truthfulness (around 2% for both bulk and milled samples). It must be noted that mean cell volume has not been altered by milling. The amount of Fe in the samples was estimated in 7(2) %wt., for bulk and 10h-BM samples.

A view to the powder morphology at mesoscopic scale is given in the SEM images of Fig. 2(a). Powder is composed of irregularly

shaped micronic particles with a broad size distribution showing a slight tendency to agglomeration. Most of particles seem to be in the range of 0.5-5.0 μm . The higher magnification micrograph of the inset reveals that particles are in fact closed packed assemblies of smaller flaky, or laminar-like, particles whose real size is difficult to establish due to the poor definition of inter-particle boundaries, but it can be roughly estimate as 100-400 nm. Accordingly, in this case the construction of a particle size distribution is nor simple, nor a reliable task. Thus, a further study of the internal structure of such micronic particles was carried out by TEM. The inset of Fig. 2(b) is a typical micrograph of the nanostructure of individual particles. The grain size distribution is typified by the histogram presented in Fig. 2(b). The average crystalline grain size, $\langle T \rangle_{\text{TEM}}$, is 27(1) nm, in admirable agreement with the $\langle T \rangle_{\text{NPD}}$ value of 24(5) nm, deduced from the Rietveld refinement [12]. As $\text{Pr}_2\text{Fe}_{17}$ is a brittle intermetallic, the reduction

in grain size is a natural consequence of progressive fracturing produced during milling process.

Fig. 3 shows the low-field $M(T)$ curve of both samples. When temperature goes through the magnetic transition region a well defined and narrow drop in $M(T)$ is exhibited by the bulk alloy leading to a TC value of 285(2) K in reasonable accordance with the reported value. Despite of its iron content, M seeks close to zero value. In contrast, the milled sample is characterised by a decrease in $M(T)$ values and a substantial broadening in the transition. The onset in the decrease of $M(T)$ starts before, the inflexion is now obtained at 292(10) K, and the magnetisation over the transition region remains relatively far from zero. In the inset of Fig. 3, the normalised $dM/dT(T)$ curves are plotted. While the bulk alloy exhibits a narrow minimum in the temperature evolution of the dM/dT , for the milled alloy this minimum is largely broadened, indicating that the ferro-to-paramagnetic transition is not well defined, probably due to the

milling-induced disorder that gives rise to a distribution of Fe-Fe interatomic distances, and then spreading out the values for the TC.

In Fig. 4 we plot the temperature dependence of the magnetic entropy change, $|ASM|$ at $\mu_0 H = 5T$ obtained from a series of $M(H)$ curves measured between 260 and 340 K. The maximum value achieved for the bulk sample, $5.7(1) \text{ J kg}^{-1} \text{ K}^{-1}$, is in good agreement with previously reported data [2,4]. The maximum is well defined and approximately coincides with the value of T_c (see Table 1). Furthermore, the milling process leads to a reduction of the maximum value for $|ASM|$ ($3.7 \text{ J kg}^{-1} \text{ K}^{-1}$), undergoing a small shift to higher temperature and a broadening in the whole temperature range (see the inset of Fig. 4, where $ASM/AS\{\max$ vs. T/T_c curve is represented). Hence, the latter must be a direct consequence of the lack of definition in the value of the T_c , shown in Fig. 3, due to a broad minimum in the dM/dT vs. T curve. Finally, it is worth

to note that even the maximum value for the magnetic entropy change decreases around 40% after milling, the temperature range in which ASM remains with more than the 90% of its maximum value is around 40 K (20 K in the case of bulk alloy). This large temperature interval with almost constant value of ASm could be interesting for magnetic refrigeration applications

Fig. 4. Temperature dependence of the magnetic entropy change $|ASM|$ at $\mu_0 H_{max} = 5T$ for bulk and as-milled Pr_2Fe_{17} alloys. The inset shows the curve normalized to $4S_j J_{jmax}$ and TC.

at room temperature using these low-cost intermetallic compounds.

Finally we can summarize the following points: (a) powders show a non homogeneous microstructure formed by flaky particles of around 100-400 nm that agglomerate forming what at a lower magnification look like larger particles, irregular in shape, showing as well a moderate tendency to agglomeration; (b) internally particles are

nanostructured with a mean grain size of around 27 nm; (c) disorder is macroscopically expressed from the magnetic point of view in the broadening of the ferro-to-paramagnetic transition, a shifting of the magnetic phase ordering temperature to higher temperatures, the reduction in the maximum magnetic entropy change and the broadening of the ASM(T) dependence.

