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Thermal Characterization of $Fe_{50}Co_{50}$ and $Fe_{65}Co_{35}$ Nanostructured Alloys

Caracterización térmica de aleaciones nanoestructuradas $Fe_{50}Co_{50}$ y $Fe_{65}Co_{35}$

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Resumen

Las aleaciones magnéticas nanoestructuradas a base de FeCo se destacan entre las aleaciones magnéticas convencionales a base de Fe por presentar óptimas propiedades magnéticas blandas, requeridas en una variedad de aplicaciones tecnológicas, industriales y biomédicas.

En este trabajo se presentan los resultados obtenidos de la caracterización térmica por calorimetría diferencial de barrido y termogravimetría magnética de polvos puros de Fe y Co, y de polvos magnéticos nanoestructurados a base de $Fe_{50}Co_{50}$ y $Fe_{65}Co_{35}$ preparados por el método de Aleado Mecánico de elevada energía.

Por medio de estas técnicas y bajo la influencia de diferentes atmósferas inertes como Helio (He), Nitrógeno (N_2) y Argón (Ar), se evidenciaron los eventos térmicos que tienen lugar desde temperatura ambiente hasta 900 °C para muestras molidas a diferentes tiempos (0, 10, 15, 20 y 25 horas), eventos tales como las temperaturas de transición magnética y de orden – desorden características de este tipo de muestras y del proceso de molienda.

Palabras clave: Caracterización Térmica, Análisis Termogravimétrico, Termogravimetría Magnética, Aleaciones Nanoestructuradas, Aleado Mecánico, Transiciones de Fase.

Abstract

Iron-cobalt based nanostructured magnetic alloys stand out among conventional magnetic Fe-based magnetic alloys because they exhibit the optimum soft magnetic properties required in a variety of technological, industrial and biomedical applications.

In this work, the thermal characterization by differential scanning calorimetry was carried out using thermogravimetric analysis and magnetic thermogravimetry of Fe and Co pure powders, and nanostructured magnetic powders based on $Fe_{50}Co_{50}$ and $Fe_{65}Co_{35}$ prepared with the high energy mechanical alloying method.

By means of these techniques and with the influence of different atmospheres as Helium (He), Nitrogen (N_2) and Argon (Ar), thermal events for samples milled at different times (0, 10, 15, 20 and 25 hours) were evidenced taking place from -70 °C to 500 °C by differential scanning calorimetry and ambient temperature up to 900 °C in the thermogravimetric analyzer. The observed events, as the temperature in the material varies, are associated with the magnetic transition temperatures, order - disorder phase transitions and stress relaxation effect which are features of this type of samples and the milling process.

Key words:

Thermal characterization, Thermogravimetric analysis, Magnetic thermogravimetric, Nanostructured alloys, Mechanical alloying, Phase transitions.

1. Introduction

The nanostructured magnetic materials have been the object of intensive study during the last decades due to the fact that they have excellent physical, magnetic and mechanical properties in comparison with the coarse-grained polycrystalline materials, which allows this type of materials to be used in different fields of application technologies (manufacture of magnetic recording sensors and technological devices), in biomedicine (transport of medicines, diagnosis and treatment of diseases) and industry, such as audiovisual mobile systems, hard drives, micro inductors, integrated transformers, magnetic sensors and micrometric-sized motors (Akkouche et al., 2011, Morán et al., 2012, Chermahini et al., 2009, Kishimoto et al., 2019).

In the search for synthetic methods of nanostructured magnetic materials that are viable and that guarantee their use for future technological applications, the most widely used physical method has been the high energy Mechanical Alloying (AM) method, which is characterized by the preparation of nanostructured alloys in the form of powders. In this research, the nanostructured magnetic materials are those based on FeCo, which exhibit soft magnetic properties, an increase in saturation magnetization, and high Curie temperatures (Chermahini et al., 2009, Dong et al., 2017).

Within the studies carried out on the soft ferromagnetic behavior of the FeCo system, it can be seen that the compositions of $Fe_{70}Co_{30}$ and $Fe_{50}Co_{50}$, have the highest value in the saturation magnetization (223 emu/g and 221 emu/g, respectively) as well as elevated Curie temperatures values (Qi Zeng et al., 2007). Also, microstructural and magnetic properties of nanostructured powders of the Fe_xCo_{1-x} system (x = 0.2, 0.3, 0.4, 0.5 and 0.7 at. %) are evidenced (Chermahini et al., 2009). Akkouche et. *al* have also studied the properties of $Fe_{50}Co_{50}$ powders synthesized by Mechanical Alloying at different milling times (36 to 200 hours). Likewise, the synthesis of the nanostructured $Fe_{60}Co_{40}$ powder was obtained by the AM method using a high energy planetary ball mill, at 380 rpm up to a time of 54 h.

The structure of the powders was examined by XRD. The morphology during milling process was analyzed by SEM, forming disordered solid solutions with crystallite sizes of 13 nm and the particle size did not exceed 3.6 μm (Bergheul et al., 2012).

In Colombia, there have been works about characterizations of Fe oxides nanostructured materials, where the AM method is most frequently used for the preparations of these materials. This way, from the AM method, the structure and magnetic properties of the system are considered (Pérez et al.,2012, Rincón et al., 2017). Similarly, the preparation of nanostructured powders of $Fe_{50}Co_{50}$ composition and their morphological characterization by SEM and DRX has been reported in (Yepes et al., 2014). It should be noted that the composition of the $Fe_{50}Co_{50}$ and $Fe_{65}Co_{35}$ nanostructured powders were formed after 8 hours of milling and the size of the crystallite reduction with the increase of the milling time. However, a lower value in the crystallite size was obtained after 7.5 nm to 35 hours of milling (Yepes et al., 2014).

From the aforementioned investigations, it was possible to demonstrate that characterizations of the FeCo system have been made using scanning electron microscopy, x-ray diffraction, and vibrating sample magnetometry techniques. However, few studies have performed thermal characterization using Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA) and Magnetic Thermogravimetry (TGM). Differential Scanning Calorimetry identifies transitions of endothermic and exothermic phases of a sample as the temperature varies. Thermogravimetry discloses the changes in the weight of a sample as the temperature is controlled. Therefore, in this work, the thermal characterization of FeCo-based nanostructured powders synthesized by the Mechanical Alloying method was carried out at different milling times, which constitutes a contribution of interest for the scientific and academic community in the area of thermal analysis of nanostructured magnetic alloys at lower temperatures and with different atmospheres.

2. Methodology

For the synthesis of the nanostructured powders of $Fe_{50}Co_{50}$ and $Fe_{65}Co_{35}$ by the Mechanical Alloying method, Fe (6 - 9 μm) and Co (2 μm) pure powders from Sigma Aldrich with 99.5% and 99.8% of purity, respectively, were used. For its thermal characterization by DSC, TGA, and TGM techniques, around 10 mg of sample was used for each measurement.

2.1. Mechanical Alloying method (AM)

To obtain the $Fe_{50}Co_{50}$ and $Fe_{65}Co_{35}$ samples by the mechanical alloying method, two different mills located in different institutions and under different conditions were used.

2.1.1. Synthesis of the $Fe_{50}Co_{50}$ composition alloy

Once the powders were weighed, they were placed in a 250 ml stainless steel container together with the balls of the same material; the diameter of the balls was approximately 10 mm and the weight balls to the powders weight ratio was 20: 1.

The milling process was done in a high energy planetary ball mill in a vacuum Fritsch Pulverisette 5. A milling intensity of 305 rpm was used and samples were prepared for times of 0, 8, 10, 15, 20 and 35 hours of milling. The milling process was paused every hour to avoid overheating (Yepes et al., 2014).

2.1.2. Synthesis of the $Fe_{65}Co_{35}$ composition alloy

The powders prepared were milled in a Spex 8000 mix/vibratory mill. A milling speed of 875 rpm was used and weight balls-weight powders ratio was 1: 1, where the weight of the ball was 6.95 g and the weight of the powders was 7 g. Both the weighting of the powders and the packaging of the

powders and balls into the container was carried out inside a glove chamber, (O_2 <0.1 ppm, H_2O <0.9 ppm), under Ar atmosphere, this way avoiding some kind of contamination. The milling stopped every 100 minutes, to avoid overheating the equipment (Echeverría K., 2014).

2.2. Thermal characterization

The thermal characterization by DSC, TGA and TGM was made at the thermal analysis research laboratory of the Autónoma de Occidente University (Cali, Colombia).

2.2.1. DSC

For the thermal characterization of the samples by DSC, a Differential Scanning Calorimeter (DSC) (Q 2000 TA Instruments) was used. This technique allows the observation of all the events associated with absorption or release of energy.

The samples studied (around 10 mg by each measurement) were heated at 10 °C/min, in a temperature range from -60 °C to 500 °C (first heating), then cooled down to -60 °C; subsequently, they underwent a second heating from -60 °C to 500 °C. The thermal characterization of all the samples was carried out under atmospheres of N_2 and He.

2.2.2. TGA

For the thermal characterization of the samples by TGA, a Thermogravimetric Analyzer TGA Q 500 TA Instruments was used, for which the samples were heated in a range of temperatures between 29.5 °C and 862.9 °C at 10 °C/min under atmospheres of N_2, Ar and He, the amount of sample used for each measurement was around 10 mg.

2.2.3. TGM

The thermal characterization of the samples by TGM was carried out in a Thermogravimetric Analyzer TGA Q 500 TA Instruments, with a magnet under the studied samples, in order to measure the variation of the magnetic force of the sample according to the temperature. The same heating and quantity conditions were used as in the case of the TGA analysis.

3. Results and discussion

3.1. Thermal characterization by DSC

The thermal characterization by DSC was performed for the Pure Fe and Co, $Fe_{50}Co_{50}$ and $Fe_{65}Co_{35}$ samples prepared by the Mechanical Alloying method under nitrogen and helium atmospheres.

3.1.1. Thermal characterization by DSC under N_2 atmosphere.

Figure 1 shows the comparison of the DSC curves of Fe, Co and $Fe_{50}Co_{50}$ alloys composition for different milling times under N_2 atmosphere. It can be detailed that when mixing pure materials in equal proportion, but with different milling times, greater tension is acquired in the alloy in comparison with the pure elements. In addition, as the milling time increases, the stress release is made more gradually, going from two anomalies to five anomalies. It is also evident that as milling time increases, the total energy that the sample releases is higher in order to reach a state of relaxation. The first anomaly observed in the curves below 0 °C can be attributed to the rearrangement of spins because they tend to be aligned when the sample is heated and, in this case, the stress relaxation processes are found above 100 °C, especially for $Fe_{50}Co_{50}$ compositions milled at different times which is consistent with that reported by Safia Alleg (Alleg et al., 2013).

3.1.2. Thermal characterization by DSC under He atmosphere.

DSC results were obtained under Helium (He) atmosphere for the pure samples of Fe and Co, $Fe_{50}Co_{50}$ and $Fe_{65}Co_{35}$ milled at different times and a heating rate of 10 °C/min.

Figure 2 shows the comparison of the DSC curves of pure Fe, pure Co and composition of $Fe_{50}Co_{50}$ for different milling times and $Fe_{65}Co_{35}$ for 0 hours (unmilled) under He atmosphere. It can be determined in it that for the first heating a small exothermic anomaly almost imperceptible above 0 °C is presented for the pure Fe, which is not evident for the Co or for the composition of $Fe_{65}Co_{35}$. This may be due to a small release of energy obtained during storage, since at this temperature no type of transition for the material is evidenced.

It can also be seen in figure 2 that anomalies below 0 °C do not show for the $Fe_{50}Co_{50}$ compositions milled at different times, this can be attributed to the high diffusivity and thermal conductivity that He presents. On the other hand, the anomalies that occur in the $Fe_{50}Co_{50}$ composition milled at different times are in the range of 140 °C to 480 °C, indicating the exothermic process that occurs for the first heating. This fact is attributed to the processes of recovery, stress relaxation, and grain growth, which are induced during the milling process, consistent with that reported by (Alleg et al., 2013).

3.1.3. *Thermal characterization by DSC of $Fe_{50}Co_{50}$ milled 10 hours under atmospheres of N_2 and He*.

Figure 3 shows the results obtained from the first heating of the sample composition of $Fe_{50}Co_{50}$ milled for 10 hours by means of the DSC technique under atmospheres of N_2 and He, and in which it can be seen that the sample under nitrogen atmosphere presents the anomalies that can be attributed to the spins ordering at low temperature (between -25.65 °C and -8.21 °C and an enthalpy (ΔH) of 0.1889 J/g), which is not observable in the DSC curve obtained under helium atmosphere for the referred sample.

On the other hand, the anomalies corresponding to the exothermic processes of release of the mechanical stresses that are induced during the synthesis process start at a lower temperature under the nitrogen atmosphere (between 128,75 °C and 240,37 °C with ΔH = 15.86 J/g, and between 316.01 °C and 381.09 °C with ΔH = 19.55 J/g), which for the same sample under the helium atmosphere (between 160.29 °C at 250 °C with ΔH = 0.6414 J/g). It can be said, from figure 3, that under the atmosphere of He, the higher the temperature is, the more liberation of stored energies takes place, taking into account the second anomaly that is observed, which occurs in the range of temperature of 326.21 °C to 450 °C with ΔH = 7,120 J/g. According to the results, it can be seen that under an atmosphere of N_2, the events associated with the release or absorption of energy in the $Fe_{50}Co_{50}$ alloy are more evident than under an atmosphere of He.

3.1.4. Thermal characterization by DSC of the composition $Fe_{50}Co_{50}$ milled 20 hours under atmospheres of N_2 and He.

Figure 4 shows the results corresponding to the first heating of the sample of composition $Fe_{50}Co_{50}$ milled for 20 hours obtained under the atmospheres of N_2 and He, by means of the DSC technique.

It is also observed that the DSC curve of the sample $Fe_{50}Co_{50}$ milled for 20 hours under a nitrogen atmosphere presents the anomalies that can be attributed to the spins ordering at low temperature (in the range between -25.02 °C and -5, 46 °C and an enthalpy value of 0.09344 J/g), which is not observed in the DSC curve obtained under a helium atmosphere for this same sample.

As in the case with the sample milled during 10 hours, the anomalies corresponding to the release processes of the mechanical stresses that are induced during the synthesis process start at a lower temperature under a nitrogen atmosphere (between 128,75 °C and 240.37 °C and an enthalpy of 5.955 J/g) than for the same sample heated under a helium atmosphere (between 139.92 °C to 200 °C with

an enthalpy 0.4416 J/g). This is attributed to the releases of stored energies when the temperatures increase consistent with that reported by Safia Alleg (Alleg et al., 2013).

Once again, a better thermal behavior of the alloy is observed in N_2 atmosphere compared to the He atmosphere, since the N_2 makes it possible to register events at low temperatures that require low energies.

3.2. Thermal characterization by TGA and TGM

3.2.1. Thermal characterization by TGA and TGM under N_2, He and Ar atmosphere

The results of the thermal characterization by TGA and by TGM of the samples of pure Fe, heated in a temperature range from 29.5 °C to 862.95 °C at a heating rate of 10 °C/min, low atmospheres of N_2, He and Ar, are detailed below.

3.2.1.1. TGA and TGM curves of the pure Fe sample.

Figure 5 shows the results of TGA and TGM of the pure Fe sample at a controlled heating regime under atmospheres of N_2, He and Ar. Such results indicate the weight variation of the sample as a function of the temperature for a range from 25 ºC to 900 ºC. Likewise, the influence of the atmosphere on the pure Fe sample is observed. Both in the TGA and TGM curves there is evidence of a greater weight increase in this order: under Argon, Nitrogen and Helium atmosphere.

The use of Helium, for example, due to its properties — high diffusivity ($1.6398x10^{-10}m^2/s$) and thermal conductivity (0.152 w/km) — as a dynamic gas provides an important means of heat transfer, tending to decrease the indicators of magnetic weight acting as an infinitely large heat sink.

In the TGA curves, weight gain of the sample can be observed from approximately 250 °C up to 900 °C, attributable to some aspects such as the reordering of spins in the material, in addition to the porosity of the sample that allows the inflow of part of the surrounding atmosphere.

On the other hand, in the TGM curves, the sample exhibits a stable behavior up to 250 °C, a temperature from which it begins to gain apparent weight. The anomalies corresponding to the magnetic transitions of the Fe can also be evidenced. The first in the temperature range of 463 °C to 586 °C, and the second anomaly occurs at 730 °C, 774 °C, and 779 °C, under the atmospheres of N_2, He and Ar, respectively. This anomaly is attributed to the Curie temperature of the sample, according to M. Khajepour and S. Sharafi theory (Khajepour et al., 2012).

3.2.2. *Thermal characterization by TGA and TGM under Argon atmosphere.*

Figure 6 shows the curves of TGA (solid line) and TGM (segmented line) for the pure Fe and the $Fe_{50}Co_{50}$ alloy milled at different times. Here the sample of pure Fe and the alloy of $Fe_{50}Co_{50}$ for TGA is stable between 25 °C and 250 °C, from which they begin to gain weight. Possibly because of the porous character of the samples, part of the surrounding atmosphere penetrates into it, a fact that can be attributed to this apparent weight gain. Another reason could be a possible superficial oxidation, because the cobalt is oxidized in its outermost layer.

In the presence of an external magnetic field, the TGM curves show stability for the pure Fe and the $Fe_{50}Co_{50}$ alloy up to 300 °C, from where they start showing an apparent weight gain.

The Fe presents two anomalies: the first at 463 °C to 586 °C and the second at 774 °C. The second anomaly occurs because the pure Fe passes from the ferromagnetic to the paramagnetic phase, which corresponds to the Curie temperature of this sample.

The TGM curves for the samples of $Fe_{50}Co_{50}$ milled at different hours, show that it presents an anomaly between 480 °C and 580 °C, which can be attributed to structural changes presented by the FeCo system, which has been reported to from 550 °C by Safia Alleg (Alleg et al., 2013) and by M.

A. Consuegra et al. (Caamaño et al., 2015). The temperature of Curie is evidenced between 572 °C and 580 °C.

3.2.3. Thermal characterization by TGA and TGM under Nitrogen atmosphere

Figure 7 reveals the results of TGA and TGM under N_2 atmosphere for the pure Co samples and the $Fe_{50}Co_{50}$ composition at different milling times, subjected to a controlled heating regime, in which the pure Co is stable between the 25 °C at 300 °C. This figure also indicates the weight variation of the samples as a function of the temperature for a range between 25 ºC and 900 ºC. Likewise, the influence of the atmosphere on the pure Co sample and the $Fe_{50}Co_{50}$ composition is observed.

In the TGA curves, it is possible to observe the weight increase of the samples approximately from 300 °C to 900 °C, which can be attributed to the reordering of spins in the material. Another reason could be a possible superficial oxidation in the outer layers of cobalt, in addition to the porosity of the sample that allows the introduction of part of the surrounding atmosphere.

On the other hand, in the TGM curves, the sample shows a stable behavior until approximately 300 °C, from which it starts to gain apparent weight, also exhibiting the anomalies corresponding to the magnetic transitions for the samples of $Fe_{50}Co_{50}$ milled at 10, 15 and 20 h with Curie temperature of 500 °C, 571 °C and 586 °C, respectively.

Conclusions

DSC characterization:

- The thermal characterization by DSC of Fe, Co and $Fe_{50}Co_{50}$ alloys composition at different milling times under N_2 and He atmospheres confirmed the influence of the milling time and also the atmosphere on the DSC curves. A greater tension is acquired in the alloy in comparison with the pure elements and also the stress release is made more gradually, so more anomalies appear.
- The anomalies are associated to the rearrangement of spins (at temperatures below of 0°C), to the stress relaxation processes (above 100 °C), and to the processes of recovery, stress relaxation, and grain growth, which are induced during the milling process (from 140 °C to 480 °C) in the form of exothermic peaks in DSC curves.
- The anomalies corresponding to the release processes of the mechanical stresses that are induced during the synthesis process starting at a lower temperature under a nitrogen atmosphere than under an atmosphere of He, confirmed the strong influence of the different atmospheres in the DSC curves of the samples.
- The order-disorder phase transition is not evidenced by this technique because this transition occurs at temperatures higher than 600 °C, a temperature that exceeds the working temperatures of the equipment.

TGA and TGM characterization:

- From TGA and TGM characterization, a higher weight gain was evidenced under Argon atmosphere than under the Nitrogen and Helium atmospheres. The weight gain of the sample can be observed from approximately 250 °C up to 900 °C, attributed to some aspects such as the reordering of spins in the material as well as to the porosity of the sample. Due to the high diffusivity and thermal conductivity of Helium, it tends to decrease the signs of the percentage of weight as magnetic force.
- The TGM characterization showed a stable behavior until approximately 300 °C, then it started to gain apparent weight, corresponding to the magnetic transitions (Curie temperature) for the samples. A high Curie temperature value for Fe was obtained in comparison with the values of the other
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- samples under study, which were 750 °C, 774 °C and 779 °C under nitrogen, helium and argon, respectively. Under nitrogen atmosphere the Curie temperature values were lower.
- The effect of the surrounding atmosphere of helium on the sample does not allow to see the molecular dynamics of the sample at a low temperature. Possibly with the N_2, the sample requires absorbing or releasing more energy to stay at the temperature of the sample.

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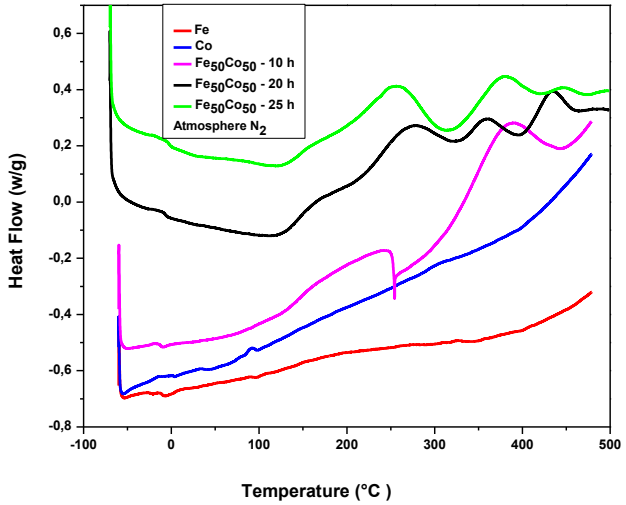

Figure 1. DSC curves of Fe, Co and $Fe_{50}Co_{50}$ samples at different milling times under N_2 atmosphere.

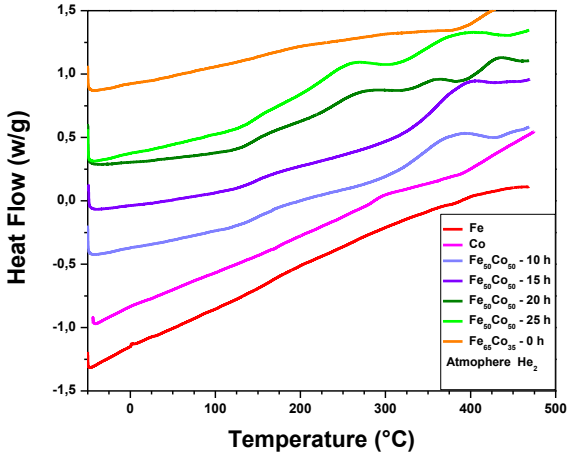

Figure 2. DSC curves of Fe, Co, $Fe_{50}Co_{50}$ and $Fe_{65}Co_{35}$ at different milling times under He atmosphere.

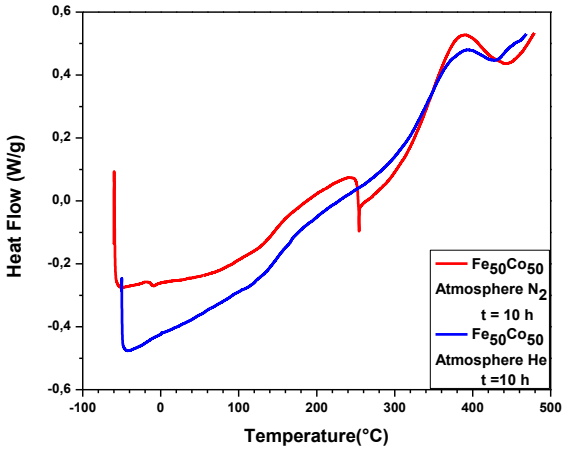

Figure 3. DSC curves of the $Fe_{50}Co_{50}$ sample milled 10 hours, under N_2 and He atmosphere in the temperature range between -60 °C to 500 °C.

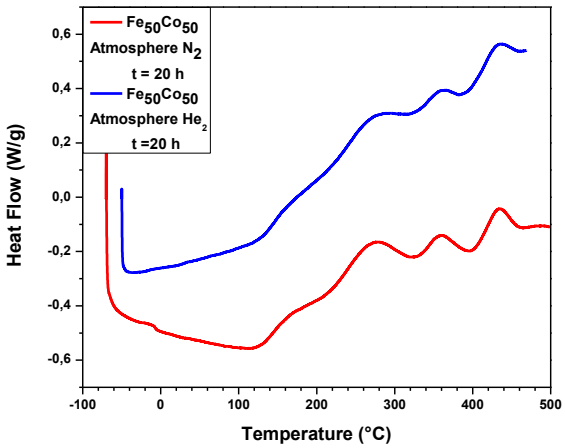

Figure 4. DSC curves for the $Fe_{50}Co_{50}$ sample milled 20 hours under N_2 and He atmosphere in the temperature range between -60 °C to 500 °C.

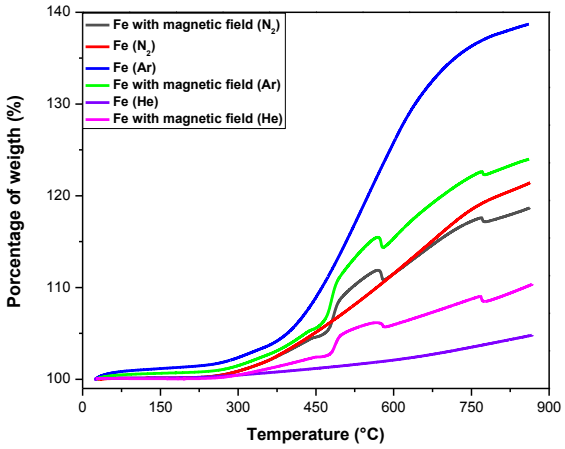

Figure 5. Variation of weight as a function of temperature for the Fe sample under atmospheres of nitrogen, helium and argon in the absence and in the presence of an external magnetic field, in the temperature range between 25 °C to 900 °C.

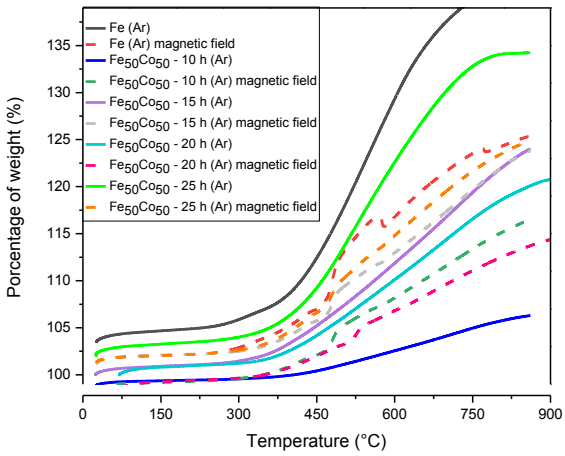

Figure 6. Variation of weight as a function of temperature for the sample of pure Fe, $Fe_{50}Co_{50}$ at different milling times under argon atmospheres in the absence and in the presence of an external magnetic field, in the temperature range between 25 °C to 900 °C.

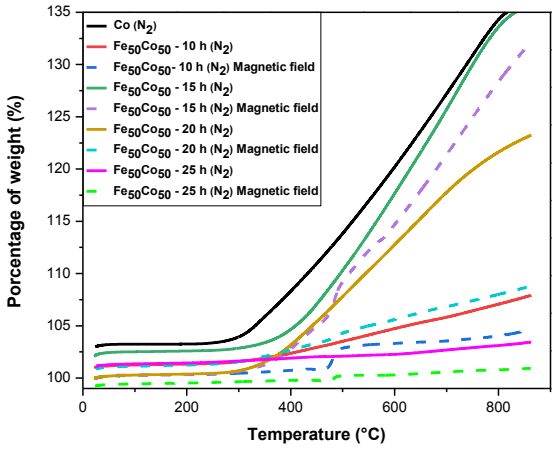

Figure 7. Variation of weight as a function of temperature for the sample of pure Co, $Fe_{50}Co_{50}$ at different milling times under nitrogen atmospheres in the absence and in the presence of an external magnetic field, in the temperature range between 25 °C to 900 °C.

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