Micro-Structure Characterization by Micro Magnetic Methods

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Abstract. The quality control of industrial components requires adaptation and the development of new material characterization and particular non destructive testing techniques. To characterize a steel, it would be useful to know its chemical composition, physico-chemical constitution, metallurgical state (annealed, hammered) and others parameters (superficial and chemical processing ...).
The testing method using Barkhausen noise (B.N.) is a particular method, which can be applied on ferromagnetic materials. It is a magnetic non destructive evaluation (NDE) method and can provide very important information on the material microstructure.
The using of the NDT method gives lot information about these nanostructures in particular the magnetic NDT techniques. In this paper, we examine the contribution of micromagnetic techniques in-the characterisation of nanostructure materials. Nanocrystalline $Fe_{(1-x)}$ Co_x , Fe ,Fe-Co-Cu mixtures have been prepared by mechanical alloying using a planetary ball mill under several milling conditions .Data analysis showed that the technique of the corrective field, the residual magnetisation, saturation moment and the eddy current was in relation with time milling of these powders(in our case Fe Ni)
***Key words*:** NDT, Barkhausen noise, FFT, remanence, corecitivity, micro defect. nanostructure

Introduction

The NDT (Non Destructive testing) by Barkhausen noise (B.N.) is a particular method, which can only be applied on ferromagnetic materials. It is a micro magnetic non destructive evaluation (NDE) method that can provide very important information about the material microstructure.The results of the here presented investigations indicate that material damage caused by micro-structural changes and micro cracking is strongly connected with changes in the micro-magnetic properties and can be correlated with the mechanical behaviour and the modification in the microstructure. Furthermore, the micro-magnetic testing method can be used for the identification of the different stages of the post-weld heat treatment process in the material.
Nanostructure matters were the subject of many studies which they constituted a new class of their genre, they represent not only a great interest in the fundamental sphere (comprehension of the physics which rules the growth, the structure and the properties of these matters), but also they offer the perspective of creating new matters with verifiable properties, usually, they are used in many applications. This last aspect is relevant for the development of new structures, for instance, the giant magnetoresistance.The common characteristic of the nanostructured matters is the variation on a nanometric scale, less than the micro structural parameters (dimension, grains size, chemical composition, atomic density, crystallographic orientation or direction).

Theory

The physical principle of this method is basing on the magnetization of material along its hysteresis loop, which is influenced by the micro structural defect stake (dislocations, grain and phase boundaries.) [2-4] which influence also steel mechanical properties. The defects temporarily break the Bloch walls movement and the reorientation of magnetic domains under external magnetic forces. The actual structure of the domains corresponds to the inner magnetic energy of the sample. This minimum can be obtained by reducing internal energy of each grain, considered as isolated from others. For each grain, it is evident that magneto static energy depends strongly its environment. The domains structure is influenced by the presence of point defects, such as atomic vacancies, interstitial, non magnetic metallic dislocation and their clusters or precipitates [7-8].
In many technical steels investigation of Barkhausen noise allows to determine the coercivity .The micro-structural parameter is derived by the peak separation of the maximum Barkhausen noise level. Magnetization is not obtained continuously but by step like and the magnetization changes are accompanied by electrical, magnetic and acoustic noise changes. With a further increase of the magnetic field strength they can over-come the pinning effects by the lattice defects inducing small electrical pulses in the micro magnetic sensor and can be recorded as the Barkhausen noise signals that can be amplified and evaluated.
It is possible to describe qualitatively the magnetization on a microscopic scale using the Bloch walls. The model of kameda and Ranjan gives the relation of the BN signal and the variation of the magnetic flux “φ “at the surface of the specimen. This variation of the magnetic density depends of the increased domain and the germination ([5]). The variation of Magnetic flow is given by the relation. $\varphi = \lambda \cdot \left(B_n N_n + \left(\delta'/\delta_w\right) \cdot N_g\right)$

λ : Coefficient of atomic magnetic moment.
B_n : Coefficient of germine domains form.
N_n : Density of germine domains.
δ' : Displacement average value of walls at increase of grains.
δ_w : Thickness of domains.
N_n : Propagation density of walls. Domains

$$d\varphi/dt = \lambda\left(B_n \cdot dN_n/dt + d\left(\delta'/\delta_w\right) \cdot N_g/dt\right)$$

$$d\varphi/dt = \lambda\left(B_n \cdot dN_n/dH + \left(N_g/\delta_w\right) \cdot d\,\delta'/dH + \left(\delta'/\delta_w\right) \cdot dN_g/dH\right) \cdot dH/dt$$

We notice that the third term is negligible in this case .This approach allows to observe two peak in the BN FFT

The hysteresis cycle represents the answer of a matter. It gives the magnetically characteristics of a sample. In a practical point of view, the hysteresis cycle can be explained by considerations of displacement and anchoring or fixing of Bloch walls in the case of a multidomain system.

Generally, to explain the hysteresis cycle of ferromagnetic matters, we use two physical grandeurs or magnitudes: the remanent or residual magnetization Mr and the coercive field Hc.
The magnetic properties of the small ferromagnetic particles were the subject of many searches. Due to the interactions of exchange, the spins of particles tend to or towards one direction according to the number and the signe of theses interactions, there is a ferromagnetic or ferrimagnetic or antiferromagnetic state. Then, a diminution of the

particles' size going with an increase of the atoms' proportion in surface has an important influence on the intrinsic magnetic properties of the matter.
In the nanostructure matters several types of interactions can existe simultaneously; namely, interactions grains-grains and interactions grains-grains'joints.

Samples

In general, the ferromagnetic properties of the investigated material are dependent on the micro-structure, the hardness and thermal treatment. Since these materials, as mentioned above ,characteristic have a strong link to the magnetic domain structure. By welding heat affected zones (HEZ) are created, representing a strong microstructure and residual stress gradient. These zones are brittle, characterized by a micro structure that depends on the preliminary operative conditions and the gradient of temperature. Also for the BN behaviour on the defects .Differents shape and dimension of the defect are manufactured on the sample

- Samples of steel grade E24 are heated at different temperature between 750°C and 1100°C without exposure time. Defects are initiated in these samples
- Samples of steel grade 45SD6 are heated at 1100°C and 850 °C. Defects are initiated in these samples.
- The technique called mechanical alloys consists generally of grinding micrometric powders of several elements or alloys to incorporate them. Th essential characteristic of this technique is to permit the obtaining of (nano-precipités or nano-objets) scattered homogeneously or heterogeneously within the matrix. The Fe,Ni,Co and Cu powders are mixed in jars filled with 16 steel tempered balls, within a controlled atmosphere (argon), the jars are fixed on the plate of the planetary grinding of the type Retsch PM400. The jars and the plate are mechanically coupled or attached. With 200 tr/min speed and varying the grinding time of each sample, the structure of the composites are determined by x-rays diffraction.

The powders densification made visible many stages. During the first to the much weak stress or strain, we take part in a putting into position of the grains; it is principally checked by the morphology of particles. The plastic deformation is produced during the second stage the roughness presence could play the part of an amplifier of strains or stress located in contact joints between the particles. On this other hand, the high proportion of grains joints let to think the mechanism of joints or similar to those intervening in the amorph matters could also take part in the deformation.

Experimental Procedure

1-Barkhausen Noise (BN) measurements [3]

The measurements were performed in an open magnetic circuit. As a consequence, there is global effective field acting on the domain walls. The samples were cycled in their hysteresis loops, excited by a slowly varying triangular wave form, 0.07Hz frequency and 200A/cm maximum field strength,. The Barkhausen signal was detected by a small yoke like inductive transducer in the central part of the sample. The signal was pre amplified by a band pass amplifier (3-250 KHz) and digitized by a TK780 oscilloscope.

2- Presentation and interpretation of results

The applied software **figure 4** allows processing of the BN by the FFT and presentation of the spectral density. For the heat treatment, we have obtained:

-The studies undertaken on the structures thermally treated show, from the amplitude peak of the FFT collected from Barkhausen noise, the influence of the temperature on the microstructure **figure1.** We can also notice that the pick amplitude decrease with the austinisation temperature .The displacement of the wall Bloch is easier due to the temperature. These results are important, it allow to evaluate an austinisation structure using the BN treated signature.
The first significance of this work is the relation study between the microstructure defects and the Barkhausen noise. We can notice that an existing defect in a thermally treated structure induces a great FFT amplitude of the BN when compared to a healthy sample **figure 2**. The sample with defect shows a very high peak of amplitude, this peak is due to the pinning of the Bloch wall by effective field
This is very significant in NDT since while controlling (a boiler for example) using BN one could differentiate between a material heat treatment and a defect due to strain
The magnetic domains are separated by Bloch walls which can be trapped by Lattice faults like foreign atoms or dislocations. The etching of grains influences inversely on the displacement of Bloch walls so their displacement is easier, the noise generated is gotten weaker and the acquisitions confirm the theoretical approach.
The second significance in this study is the confirmation of Kameda's model which is the two peaks amplitude development of the Barkhausen noise.

In fact, the free mean path of the magnetic domain depends on two parameters which are the grain size and the attendance of impurities, in our case the microdefects which induces walls slowing down movement.

In the **figures 3 (a,b and d)** below, we have represented the important parameters of hysteresis cycles of the grinded powders hour applying an outer field rather important for the saturation of our samples.
The coercive field and the residual magnetism were deduced from hysteresis curves **figure4**, they are represented according to grinding duration. We remark in Hc and Br 2 zones, the first between 0 hour and 8 hours, below 8 h an increase Hc and Br, our matter becomes magnetically hard due to the refinement of the iron and nickel grains, plus the size (ferromagnetic)reduces or brings down the increase of Hc,Br
A progressive decrease between 8 and 12 hours that can be interpreted by the formation of $Fe_{80}Ni_{20}$ alloy which is confirmed by the X-rays results.
The second between 12 hours and 100 hours, an increase Hc Br with an increase of grinding time because of the size, we know that the nickel has a non ferromagnetic behaviour into an iron alloy.
The same result is obtained by eddy current measure **figures 3(f)**

The $Fe_{80}Ni_{20}$ structure varies according to the grinding time. The iron x-rays' diffraction reveals a broadening or widening of peaks at the plane levels (110),(200),(211) and (220). The peaks intensity of nickel decreases till its total disappearing in 12 grinding hours.
The $Fe_{80}Ni_{20}$ solid solution is formed by substitution of centred-sided nickel of cubic tracery by the centred cubic tracery iron, this solution is called camacite (centred cubic tracery)

Conclusion

The combination of different techniques of characterization (micromagnetic measures and eddy current permitted to conclude clairly the following scenario or background
The most significant conclusion is the influence of the type, and dimension of defect on the BN signal analysis. The study of BN by digital processing can give more information about the defect (type, position, dimension…). . These results are very important, it's possible to determinate anomalies such dislocation and micro defects…
The most important result obtained through this work, is first the relation between micro defect and the BN. The defects BN signal is different according to its shape, its dimension and its position with regard to the surface.
Evaluation of welds is extremely important for the materials performance; hence the quality and the integrity of these joints must be controlled, to ensure that no problem exists in weld. Steels chosen are commonly used in industrial field and often submitted to thermal processing such as welding operations. The results of the peak amplitude of the FFT with exposure time gives an aspect on the characteristic structure of the specimen... For the same structure, we can evaluate the structure, Finer structure or smaller grain sizes increase the density of available pinning sites, reducing domain wall motion and therefore an increase of the BN . Both micro magnetic parameters show a good sensitivity with respect to micro structural changes during thermal processing and thus this non-destructive method can be used to characterize thermal treatment in a fast and easy way.
The residual, the coerecitivity,the saturation moment and the eddy current methods can give the most result about the nanostructure determination.
The morphology of the nanostructure powders can be described by an assembly of nanocrystalline grains directed at random, knitted to each other by joints of grains. The exploitation of the experimental results was leaded jointly and has permitted a microstructural average description of these powders. The nanostructure powders are then constituted of crystallized grains of a size about 13nm parted by a small thickness of the grains'joints. In other respect, the use of these techniques permitted a comparison of experimental results and knowledge of structural and magnetic properties of nanostructure powders. This permitted to arrive to a conclusion of an average microstructure and an average magnetically behaviour of powders.

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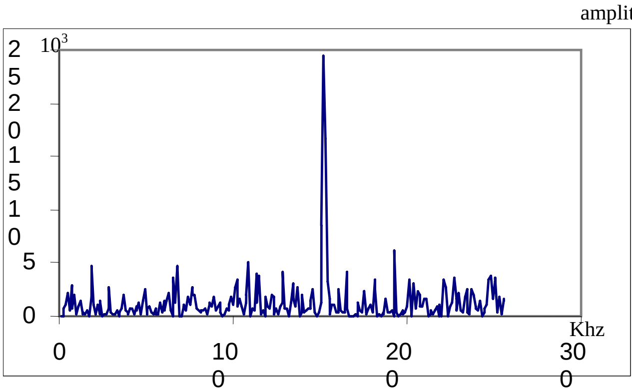
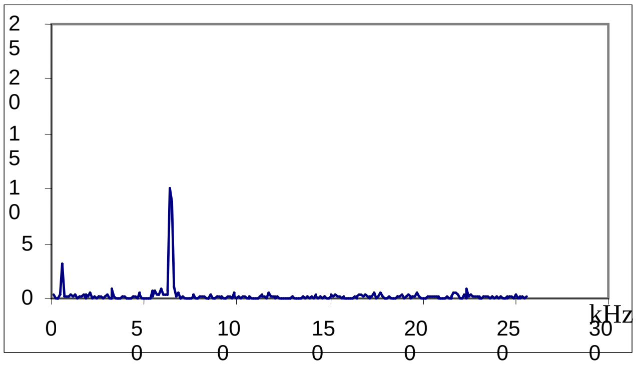
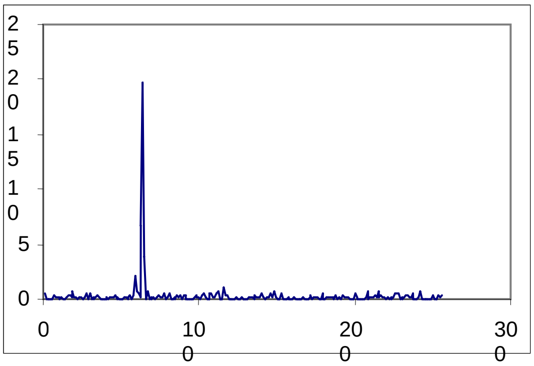


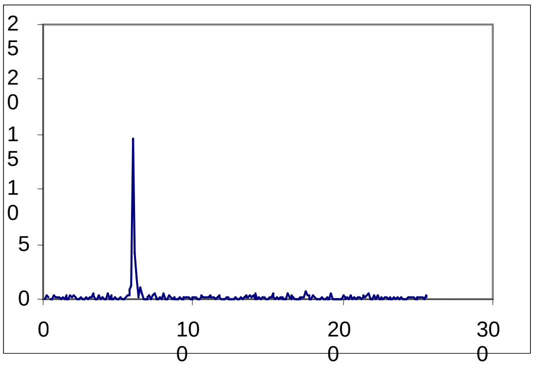
spectral density of E24 non treated



Spectral density of E24 at 750°C



Spectral density of E24 at 850°C



: Spectral density of E24 at 1100°C

Figure1 :Amplitude spectral density of E24 heated

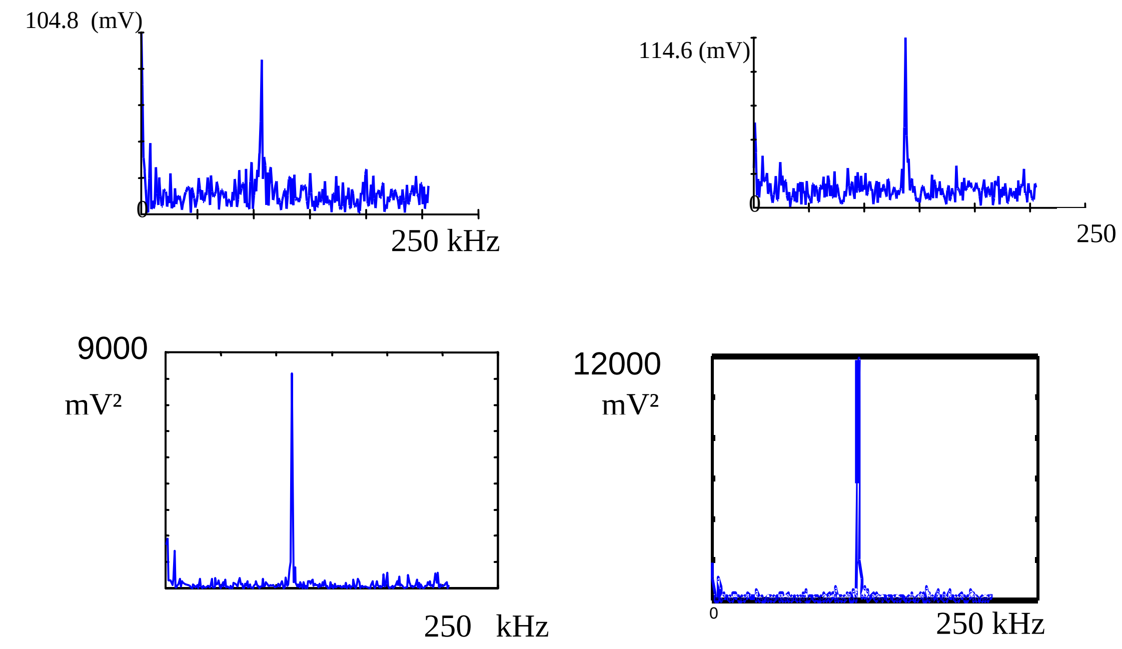
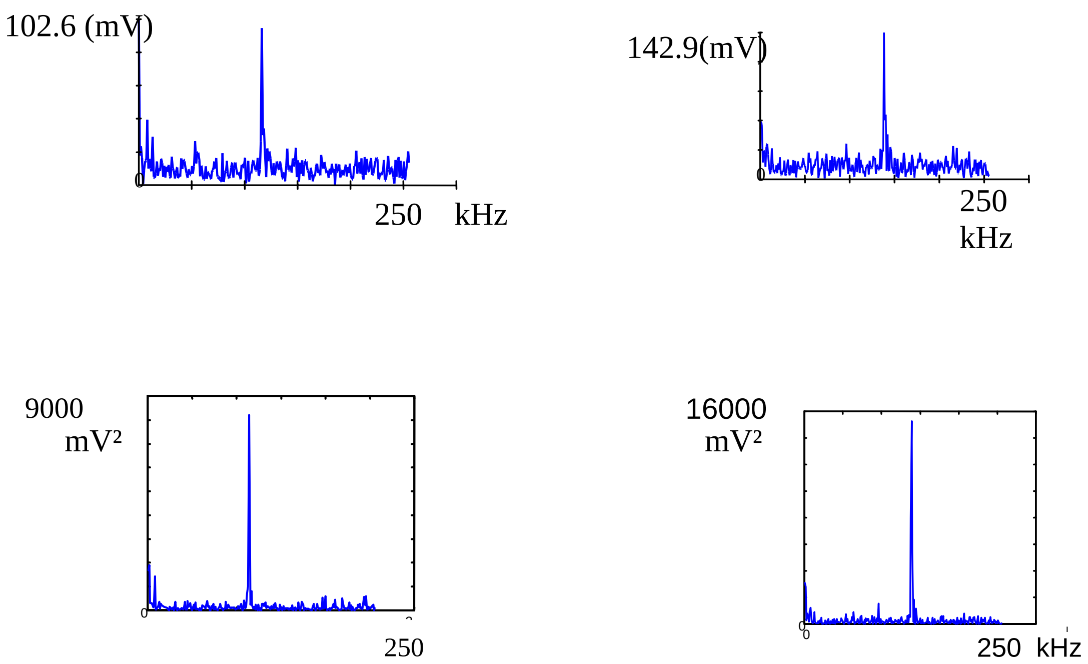


Fig2 FFT and spectral density of45SSCD6

without defect

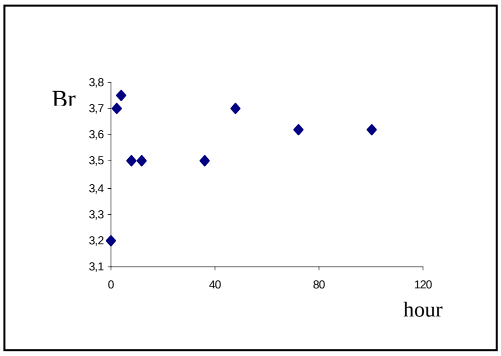
With defect



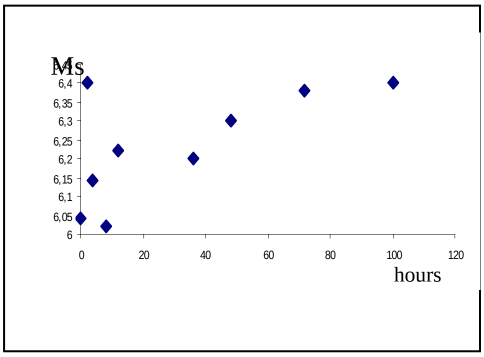
without defect

With defect

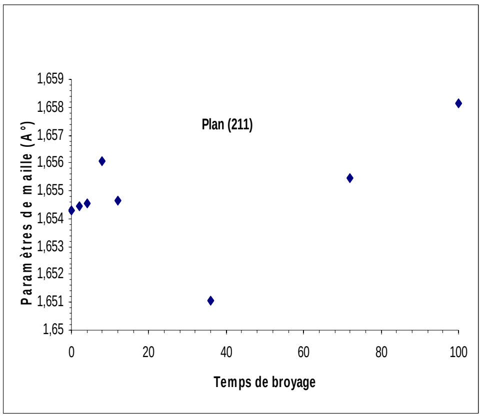
Fig 2:FFT and spectral density of 45SCD6 treated at 1200°(1,5h)



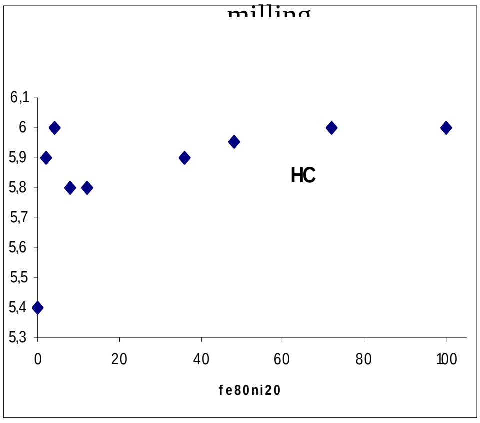
a:Remanence according to the time



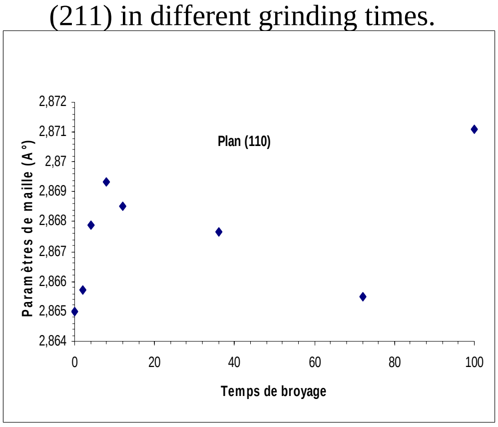
b:Saturation moment according to the time milling



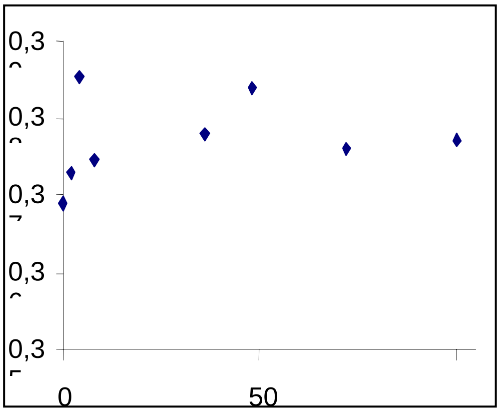
c:Crystallography parameters in plane (211) in different grinding times.



d:Coercitivity according to the time milling



e: crystallography parameters in the plane (110) in different grinding times.



f: Impedance according to the time milling

Fig3 :Micromagnetic methods and cristallography as a function of milling time for Fe_{80} NI_{20} alloy.

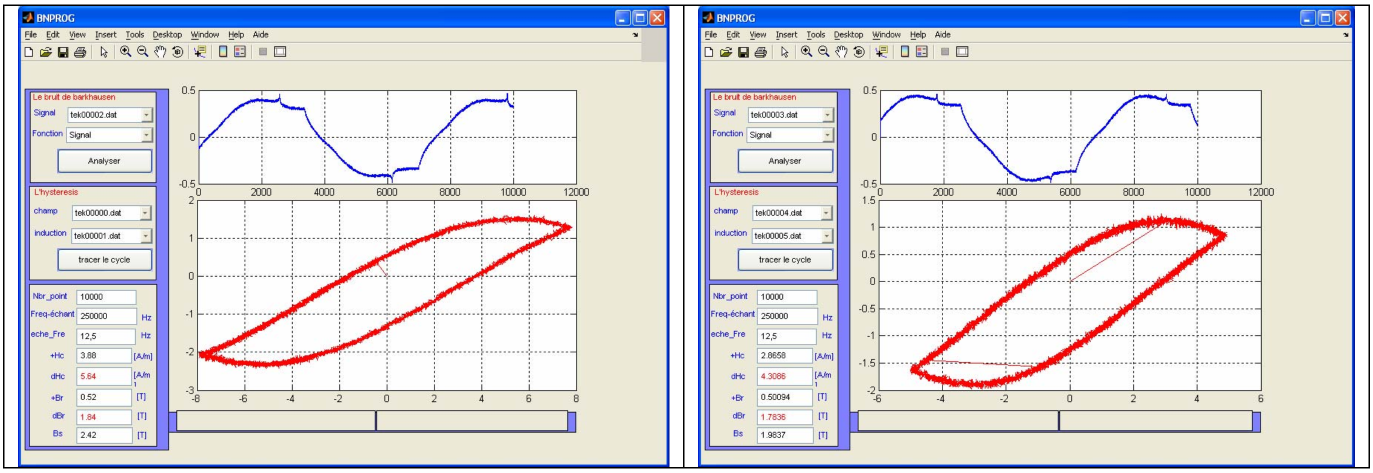


Figure4 : loop hysteresis and BN treated by Software