COST 526 – Project *CZ4*

Final Report

Optimization of heat treatment of magnetic materials applying the thermomagnetic curves data

Tomáš Žák

Institute of Physics of Materials, AS CR

Žižkova 22, CZ-61662 Brno, Czech Republic

1. Introduction

To produce modern magnetic materials the rapid cooling from the melt is used very often. The as-prepared material is in an amorphous or microcrystalline form and its optimal properties can be reached after a specific heat treatment. This process results in irreversible changes in the structure and orientation. As a typical example, there are the nanocrystalline materials gained by controlled crystallization from original iron- or nickel-based amorphous alloys using various techniques. During their subsequent heat treatment, only limited changes in the material structure are admitted. Usually they include some stress relief and building of fine crystalline structure or precipitation, but almost in no cases phase transformations, grain coarsening or recrystallisation.

For finding the critical temperatures and classification of these processes, thermomagnetic curves can be used [1, 2]. Examples of thermomagnetic curves are presented in Figs. 1 and 2 for two different materials.

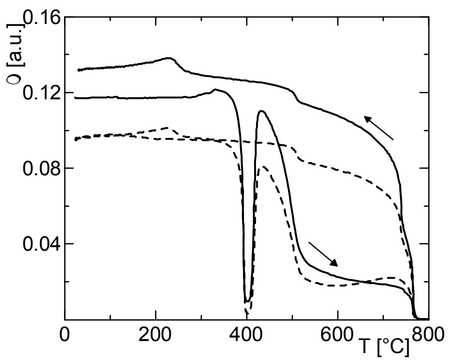

Figure 1: Thermomagnetic curves of two pieces of the same material (amorphous Fe-B alloy), irreversible changes are evident.

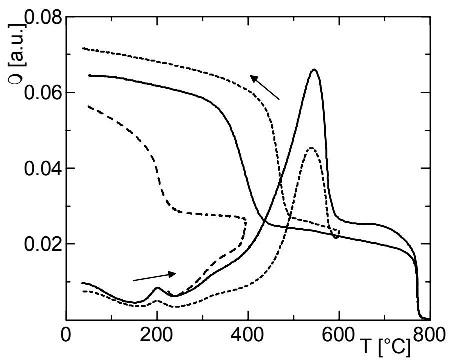

Figure 2: Thermomagnetic curves of iron oxides that differ by the reached temperature; only the solid line curve crossed the Curie point.

Thermomagnetic curves mean a graphic representation of the sample magnetic moment vs. temperature dependence. The appearance of such curves can be very diverse; however, in case of a ferromagnetically ordered matter, there is always present at least one steep descent connected with the Curie temperature of the material, and the sample above a certain temperature (also called Curie point) is no longer magnetically ordered. However, also for some other types of magnetic ordered, appropriate critical temperatures are clearly visible in most cases. Thermomagnetic curves are usually measured for both increasing and decreasing temperature. Difference between these two branches of measured dependence can serve as an evidence of irreversible changes in the material.

2. Goal of the project

The aim of the work is to enable finding a position of all the critical temperatures of the analysed material as precisely as possible and distinguishing the character of their origin. This knowledge will allow us to optimize the heat treatment process of perspective amorphous and microcrystalline alloys.
As most promising, there was found the process, where the raising-temperature-branch of the thermomagnetic (TM) curve is measured and subsequently its derivative is computed. The measurement itself is very simple; the derivative curve is easy to produce. Although the measured curves seem to be quite smooth, it is obvious that the derivative process makes to be pronounced not only the parts of the curve that have their physical meaning, but also many artefacts.
In addition, the process of computing the derivative itself can be done in several different ways. Because of final steps and numerical instabilities, the level of noise originated consequently can be very different and in some cases too intensive. Under unfavourable circumstances, also here artefacts without physical meaning can be generated. To illustrate these problems, Figs. 3 and 4 were prepared.

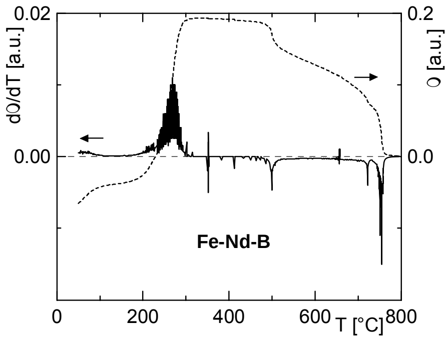

Figure 3: Extremely high level of noise on the derivative of an apparently smooth curve as consequence of a numerical treatment.

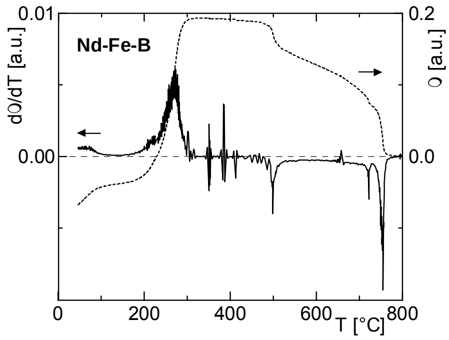

Figure 4: Artefacts placed on positions where the thermomagnetic curve exhibits no physical processes.

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Because of just described problems, it is obvious that usage of some processes of smoothing or filtering will be inevitable also in case of high quality measurements. As one of the possible ways of the noise problem solution, was filtering using an artificial neural network (ANN) [3] – see also the chapter 6. The obtained signals were very close to the original shapes and the widths of the most important shapes were almost unchanged. Less important parts of the curve were suppressed during the de-noising process.
The results obtained using these filtering algorithms were compared to the results of the sliding weighted average approach, wavelet analysis or cubic spline smoothing. Each of these methods returned very similar results, but the cubic spline algorithm seems to be the most adequate approach for our needs. The advantage of this technique is the possibility of direct derivative calculation of the measured curve [4]. An example of such a satisfactory result of data filtering (curve smoothing) can be seen in the Fig. 5.

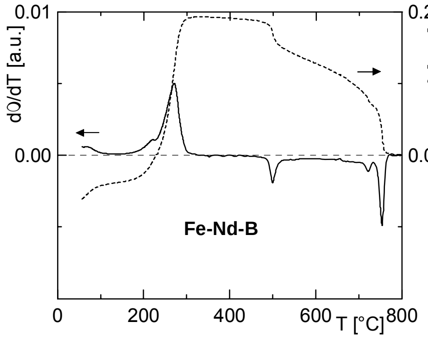

Figure 5: Nearly ideal result of smoothing (filtering) when using cubic spline algorithm with suitable smoothing parameter value.

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It also allows the control of smoothing efficiency, which can be very important when curves contain narrow peaks of various intensities.

3. Simulator, calibration, quality function and optimization algorithms, including assessment with respect to alternatives

After successful filtering (smoothing), the modelling of the thermomagnetic curve can be performed to automate the process of gaining critical temperature values. In this study, the parametric modelling of the thermomagnetic curve's derivative was applied, supported by the manual setting of peak positions at first. The exact peak positions, intensities, and widths were obtained by fitting the suggested model using the least squares method. A precise analysis of the results allowed the distinction of critical temperatures for the chosen materials. Preliminary, the Lorentzian shape of the temperature peaks was employed for the derivative curve. Thus according to the peaks positions, various processes can be distinguished through their critical temperatures (Fig. 6). Moreover, there is an idea, that the width and intensity of lines as appeared on the derivative curve can help us to distinguish the character of individual processes taking place in the material during heating.

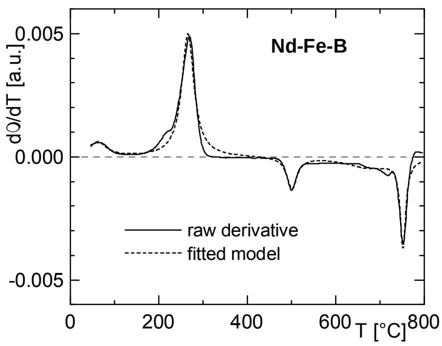

Figure 6: Comparison of the raw smoothed derivative curve with the fitted model giving the exact values of critical temperatures.

The main disadvantage of the above-mentioned approach is the necessity for human interaction, when constructing the model and setting the initial values of parameters. To avoid this problem, we have designed an algorithm used for the estimation of the preliminary line positions, intensities and widths. Our solution is based on the tracing

of the derivative curve behaviour that illustrates the character of the measurement main curve. The most important advantage is the ability of the algorithm to distinguish between critical temperature peaks and artefacts.
Additionally, the program for algorithmic estimation of peak positions was implemented and tested on several thermomagnetic curves of various materials. The processing with estimation of the main critical temperatures returns us the precisely determined positions and allows further analysis of the material features.
Fig. 7 presents the parametric least squares modelling of the thermomagnetic curve's derivative with an algorithmic estimation of the peak positions.

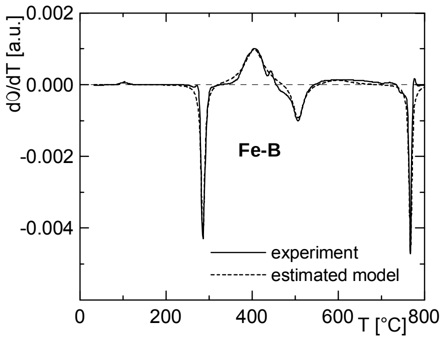

Figure 7: Comparison of the raw smoothed derivative curve with the preliminary model as estimated by the implemented algorithm.

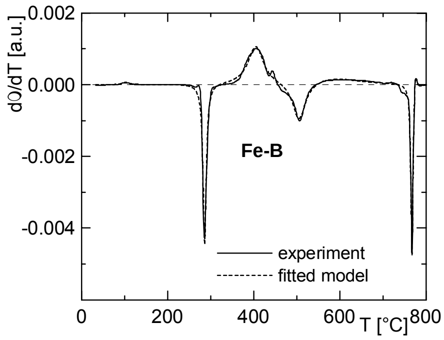

Figure 8: Comparison of the raw smoothed derivative curve with the fitted model as preliminary estimated by our algorithm.

Result of the fitting of model gained from the algorithmic estimation is in the Fig. 8. You can see that there is no substantial difference between them and the algorithm for preliminary parameter estimation is satisfactory on the first glance.
When studying the results of model parameter estimation algorithm more precisely, some imperfections can be detected, especially in case of minor and overlapping peaks. We suppose avoiding this problem taking into account also second derivative values.

4. Main scientific outcome

Detecting the shape of the thermomagnetic curve for raising temperature, we are able to gain without other human interaction values of most important critical temperatures of processes occurring during heat treatment and partially also distinguish nature of these processes. From the scientific point of view, these results can be confronted with other methods that are able to detect structural changes and changes of phase composition of material studied, e.g. X-ray diffraction. Such complex information is very important for the further development of amorphous and microcrystalline magnetic materials and for explaining their behaviour.

5. Main technical outcome

The gained results give us a survey of all necessary properties important for future treatment of the measured material. The accordingly treated material can be tested on the awaited properties afterwards; there can be measured once more thermomagnetic curves and the heat treatment can be modified several times.

Thus, in spite of still not perfect algorithm for preliminary estimation of model parameters, we succeeded in building an automated treatment of thermomagnetic curves when finding optimized heat treatment of amorphous and/or microcrystalline alloys.

6. Collaboration within COST 526

Concerning the problems with suitable way of filtering of thermomagnetic curves derivative, cooperation was offered by colleagues from the Faculty of Metallurgy and Material Science, Department of Computer Methods in Metallurgy, University of Science and Technology in Kraków, Poland, particularly by Dr. Łukasz Rauch. They were able to apply an alternative way of treating of derivative curves using an artificial neural network (ANN).
Fig. 9 presents the comparison of three various approaches in solving the problem of filtering.

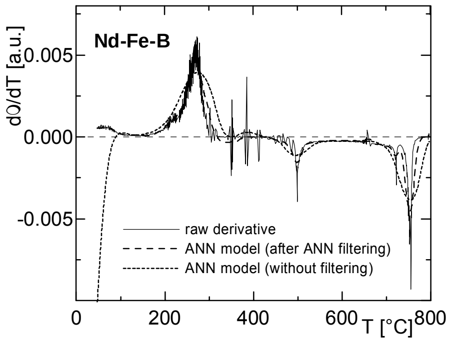

Figure 9: Comparison of various pre-processing methods based on artificial neural network.

7. Cooperation with industry

The method is still on the laboratory level and has not been developed to the point in that it can be immediately implemented in the industrial field. There was no company directly involved into the project.

8. References

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