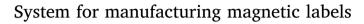
Contents lists available at ScienceDirect

## Measurement

journal homepage: www.elsevier.com/locate/measurement



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#### ARTICLE INFO

Keywords: Magnetic labels Annealing Amorphous material

#### ABSTRACT

As a result of this study, a device has been developed for the pulsed current annealing of amorphous magnetic materials covered with non-conductive dissipative material, such as silicone oil. This system makes it possible to obtain magnetic labels using a single material, thereby reducing production costs and preventing the material from rusting during the treatment process. Furthermore, maintaining the sample in tension throughout the process prevents it from becoming deformed or breaking during the heat treatment. Lastly, it has been demonstrated that the labels obtained can be detected by measuring the spectral response.

#### 1. Introduction

The excellent magnetic properties of amorphous materials arouse great interest from a technological point of view, due to their use in sensors and security systems [1].

Since the appearance of the first amorphous metal in 1960 [2], the study and development of amorphous materials has represented a field of great interest due to their basic characteristics, such as the lack of translational periodicity and the non-directional nature of their bonds. This constituted a major departure from other materials known at the time.

One of the fundamental characteristics of amorphous materials is their soft nature. The large amount of compositions that exist and the option to vary their properties through various treatments, make it possible to design the most suitable material for a given application. Starting with annealed amorphous ferromagnetic samples, their properties can be changed locally to obtain the most suitable type of material for use in the field of sensors and magnetic labels [3–6].

Some of the magnetic labels that exist on the market can be deactivated (on taking a product out of a shop, for example) or activated (on returning a book to the library, for example). These kinds of labels are composed of a base made of soft magnetic material, with several pieces of hard magnetic material put on top of it. That way, the stray field of the hard magnetic material inhibits the magnetisation of the soft part, such that if the hard material is demagnetised, the label remains activated. This method implies the use of two different materials and requires a complicated and expensive manufacturing process. These disadvantages could be addressed if it were possible to obtain materials which featured alternate soft and hard areas. In addition to the aforementioned advantage of using a single material, the manufacturing speed could be high enough to warrant its use in industrial processes [7].

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Due to the metastable state of amorphous materials, heat treatment would result in significant variations in their characteristics. Since heat treatments must be applied in annealing processes in order to reach different temperatures and be able to study heat transfer processes, various research projects have focused upon the design of the furnaces so that their energy consumption is as low as possible [8,9].

Various studies have been carried out concerning the influence of heat treatments on amorphous materials, and it has been proven that annealing reduces the internal tensions of such materials and that the aforementioned treatment may be used to produce a uniaxial magnetic anisotropy in the samples. In 1981, Hasegawa et al. [10] used a conventional furnace for the heat treatment of  $Fe_{75}Ni_4Mo_3B_{16}Si_2$  nuclei, demonstrating that it was not possible to minimise the coercive field and hysteresis loss at the same time.

However, using other heat treatments, it is possible to minimise both the coercive field and the losses. In 1984, Taub [11] pointed out that uniform heat treatment of amorphous materials, so that they may be used as transformer cores, is difficult to perform due to the poor heat conduction of the core. He also created a model to explain and predict the flow and structural changes which take place in amorphous materials at high temperatures. He subsequently argued that the optimum heat treatment of amorphous materials, involves applying a very high temperature for a short space of time, in other words, by means of rapid heating and cooling. For such treatment, Taub used the amorphous material Fe<sub>81'5</sub>B<sub>14'5</sub>Si<sub>4</sub> putting it in thermal contact with a quartz furnace at a temperature of 500 °C for 45 s, successfully reducing the

https://doi.org/10.1016/j.measurement.2017.11.021

Received 10 May 2016; Received in revised form 13 September 2017; Accepted 9 November 2017 Available online 10 November 2017 0263-2241/ © 2017 Elsevier Ltd. All rights reserved.



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coercive field and the material losses.

At the same time, in 1983, Lanotte [12] performed the annealing of strips of Fe<sub>40</sub>Ni<sub>40</sub>P<sub>14</sub>B<sub>6</sub> using a continuous-wave laser with a diameter that was six times larger than the width of the strip, placing the entire system in a helium atmosphere in order to prevent rusting. The temperature of the sample is calculated using a PtRh-thermocouple placed in contact with the non-irradiated side of the sample, ensuring a measurement error in the range of about 1%. Laser annealing is done for periods of 1 s, comparing the results with those obtained from conventional furnace annealing for a period of 2 h. The results obtained showed that at temperatures of below 500 °C, laser annealing causes a relaxation of internal tensions in the range of about 30% greater than that achieved by annealing in a conventional furnace. They also produced a temperature model, in which they calculated the behaviour of the temperature using a dual numerical integration method, and experimentally obtained the variation of the permeability of the MET-GLAS 2826 alloy according to the external tension applied.

In turn, Jagielinski [13] carried out the annealing of amorphous materials by passing an electric current through them for a short space of time (Flash-annealing). He applied this method to the zero magnetostriction amorphous alloy  $Fe_{4.7}Co_{70.3}Si_{15}B_{10}$ , by having a maximum electric current of 5 A passed over samples measuring 5 cm in length for periods ranging between 0.1 and 20 s; achieving a maximum temperature of 800 °C without the sample crystallising. As such, he obtained a maximum permeability value, higher than that obtained through conventional annealing, in turn reducing the anisotropy caused during the cooling process and preventing surface rusting, such that the electrical properties of the material did not deteriorate. The main problem encountered in this project concerned establishing the temperature of the sample. Not being able to measure it directly, Jagielinski resorted to estimating it based on the time taken to reach the Curie point.

Continuing the work of Jagielinski, Gibbs et al. [14] applied flashannealing to  $Fe_{40}Ni_{40}B_{20}$  (VAC 0040) and  $Fe_{40}Ni_{40}P_{14}B_6$  (METGLAS 2826), studying the variation of the coercive field according to the number of pulses. They used samples measuring 10 cm in length, through which they passed currents of up to 10 A for periods ranging between 0.1 ms and 112 ms, achieving a maximum temperature of 500 °C. Using the aforementioned treatment, they obtained a coercive field for METGLAS 2826 of 0.55 A/m and for VAC 0040 of 0.65 A/m, in contrast to the result obtained by annealing in a conventional furnace, of 0.9 A/m and 0.75 A/m for both samples respectively. Using 10 ms pulses they achieved a temperature of 500 °C, estimating the formation of 10<sup>16</sup> nuclei/m<sup>3</sup> of crystallisation, each nucleus subsequently growing into a crystal measuring an average of 0.1 µm in diameter. However, annealing in a conventional furnace at 375 °C for 300 s produces 10<sup>17</sup> nuclei/m<sup>3</sup>, each one growing to an average diameter of 0.3 µm.

The effect of the crystals on the magnetic properties depends on the size of the crystal in relation to the width of the wall, and on the dispersion of the crystals in relation to the geometry of the domain. In a Fe-rich amorphous material, eliminating internal tensions through annealing the average uniaxial anisotropy is in the range of about 100 J/ $m^3$  and the exchange constant is approximately  $3 \cdot 10^{12}$  J/m, giving a wall width in the range of  $0.7 \,\mu$ m. As can be observed, this is comparable to the size of the crystals produced in oven annealing, producing an increase in the nucleation centres and an increase in the coercive field of the material. As such, by means of flash-annealing, a significant reduction in the coercive field is achieved compared to conventional heat treatment, and furthermore the nucleation and growth of crystals is prevented and the degree of simple rusting reduced.

As can be seen, the most optimum heat treatments are current and pulsed current annealing [15,16]. The disadvantage of these methods is that it is not possible to determine the real temperature that the material reaches during the treatment. The method developed as part of this research manages to obtain this fundamental parameter in heat

treatments. As such, a study of temperature dispersion has been carried out using amorphous strips, demonstrating that despite the small size of the sample, it is possible to measure the distribution of the local temperature [17].

Furthermore, studies have been carried out to detect the tension applied to ferromagnetic material using Magnetic Barkhausen Noise (MBN) [18], as have various projects related to the production methods used for magnetic labels [19,20]. For example, in [19] a heat treatment system is presented, using materials whose main constituent is iron, based on a conventional furnace immersed in a system with a low oxygen content, so as to avoid the issue of the material rusting, which occurs during conventional annealing processes. With this kind of treatment, magnetic labels have been obtained by inducing several anisotropies during the annealing process.

One of the main issues encountered when producing magnetic labels using annealing is the material rusting. One way of preventing rusting during the manufacturing of labels involves using short annealing times. As such, the patent [21] describes a device and a method used for the local annealing process of the material using laser pulses lasting milliseconds.

In the patent [16], the authors of the present paper presented a local annealing system using pulses of electrical current to obtain magnetic labels which can be activated and/or customised.

This paper presents an alternative method which on the one hand circumvents the issues associated with using large and expensive facilities, and on the other hand resolves the problem of possible rusting during the annealing process. Our device permits pulsed current annealing of the material within a dissipative material such as oil, which enables magnetic labels to be obtained [11,12].

#### 2. Experimental method

Generally, the current annealing of amorphous magnetic materials is done homogeneously using conventional furnaces. Using the device developed as part of this research, the intention is to carry out nonhomogeneous annealing. As such, the samples have been covered with a dissipative material, so that the nucleus of the sample can be crystallised, whilst the surface remains in an amorphous state. This material, which will help to dissipate the heat from the surface of the sample, should not be an electrical conductor. Of all of those tested, that which produced the best results was silicone oil.

In order to keep the amorphous material sample immersed in a cooling liquid, whilst it is subjected to pulses of electrical current which make the annealing process possible, a specific device has been designed for that purpose.

### 2.1. Device description

The device consists of an element that enables the sample to be adequately positioned in a cooling cuvette containing silicone oil.

The device consists of a support (Fig. 1) and a cooling cuvette (Fig. 2). The support (Fig. 1) must be made from material that is a thermal and electrical insulator. It is made up of a fixed T-shaped part (3) with three perpendicular arms (one placed on either end of the short side of the T and another halfway up the long side) and a moving part (5). Both are joined by means of a cylindrical shaft (6), which enables the moving part to rotate (5) in relation to the fixed part (3). Stuck to the inner side of the moving part (5) there is a weight (7) the purpose of which is to tense the amorphous material sample (9), with a view to avoiding the forces created by the current passing through it, which could distort it. This tension is produced due to the weight (7) being stuck to the moving part (5), thereby displacing the centre of gravity of the whole in relation to the axis of the shaft (6), which generates a force couple and the aforementioned rotation of the moving part (5) in relation to the fixed part (3) around the shaft (6). In the base of the arm perpendicular to the long side of the T and in the base of the moving

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