# Thermal annealing of soft magnetic materials and measurements of its magnetoelastic properties

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Abstract: Paper presents both methods of the most advanced thermal annealing as well as available methods of testing the magnetoelastic properties of soft magnetic materials for technical applications. Selected features and conditions important for annealing of ring-shaped cores made of the magnetoelastic amorphous ribbons are described and an example of thermomagnetic processing is shown. Unified methodologies for testing of magnetoelastic properties of the frame-shaped and the ring-shaped cores, for both compressive and tensile stresses are presented.

**Keywords:** soft magnetic materials, thermal annealing, magnetoelastic properties

S oft magnetic materials are widely used in different technical applications, as the cores of inductive components [1] recently mainly for switching mode power conversion [2], power transformers, current transformers [3] and surge protectors as well as cores of magnetomechanical sensors [4]. In all these applications both amorphous and crystalline soft magnetic materials can be applied. However, both these materials have to be subjected to thermal annealing [1], until they achieve required magnetic properties.

Influence of different modes of thermal annealing on magnetic properties of soft magnetic materials is recently intensively studied from both theoretical and technical point of view [5]. On the other hand, these studies are focused mainly on changes of magnetic properties during annealing, whereas increase of magnetoelastic stress sensitivity is often neglected. In technical applications, stress sensitivity of magnetic materials may be very crucial. First of all, due to miniaturization of magnetic components, even relatively small forces applied during assembling process may generate significant mechanical stresses in the cores of inductive components. This may lead to decrease of its permeability and increase of the core losses [6]. As a result high stress sensitivity of the core may lead to malfunction of the device due to overheating of the inductive element.

Presented paper is trying to fill this lack. It presents both methods of the most advanced thermal annealing as well as available methods of testing the magnetoelastic properties of soft magnetic materials for technical applications. As a result it may be the base of development of unified methodology of thermal annealing in industrial scale with control of magnetoelastic properties of soft magnetic materials.

## 1. Influence of thermal annealing on functional properties of soft magnetic materials

Magnetoelastic Villari effect is connected with the changing of the total free energy of the magnetic material under the influence of stresses caused by external forces. The total free energy E of a magnetized sample may be presented as a sum of the individual free energies [7]:

$$E = E_{H} + E_{D} + E_{R} + E_{\sigma} + E_{W} \tag{1}$$

where:  $E_H$  is the energy of the magnetising field H,  $E_D$  is the energy of demagnetization of the sample,  $E_R$  is the random anisotropy energy,  $E_{\sigma}$  is the magnetoelastic energy and  $E_W$  is exchange energy. The magnetoelastic energy  $E_{\sigma}$  is given by [8]:

$$E_{\sigma} = \frac{3}{2} \lambda_s \sigma \sin^2 \phi \tag{2}$$

where  $\lambda_s$  is the saturation magnetostriction and  $\phi$  is the angle between magnetisation  $M_s$  and the direction of the stress  $\sigma$ .

The magnetoelastic sensitivity is connected with participation of magnetoelastic energy  $E_{\sigma}$  in the total free energy E of the sample. If this participation is increases, stress sensitivity increases as well. Due to the fact, that thermal annealing reduces residual stresses in the sample, it increases participation of magnetoelastic energy  $E_{\sigma}$  and leads to increase of stress sensitivity. Moreover, the nearly zero magnetostrictive magnetic materials, (such as cobalt based amorphous alloys) are also stress sensitive. It is caused by the fact, that saturation magnetostriction  $\lambda_s$  of

the material is stress dependant [9]. As a result nearly zero magnetostrictive materials exhibit significant magnetostriction under mechanical stresses [10].

### 2. Developement of methodology of thermal annealing

Rapidly quenched materials in form as the ribbons are almost never used as soft magnetic materials in asquenched state - the suitable thermal or thermomagnetic treatment, (i. e. simultaneous application of both thermal exposure and suitably oriented external magnetic field), is necessary to optimize or tailor selected magnetic parameters and to stabilize them. This holds even more strongly for magnetic circuits from nanocrystalline rapidly quenched alloys, (where the optimal nanograin size and content are achieved through controlled transformation from amorphous state as a necessary), yet not sufficient, prerequisite for obtaining high-performance soft magnetic properties. The effects described above, especially the magnetoelastic sensitivity, have to be taken into account. Additionally, application of external magnetic field during thermal treatment can selectively enhance important magnetic characteristics of the treated material [11].

In order to be able to control the thermal treatment of the cores magnetic areas, especially with non-negligent mass (exceeding several grams), a special furnace has to be used. Such a furnace has to be able to allow ramping to or from selected isothermal annealing temperature with rates ranging from 0.1 to about 10 K/min, has to have temperature stability typically better than 1 K.

At the same time it has to allow for reasonably small yet controlled thermal gradients imposed onto the annealed magnetic circuit. In order to minimize stresses due to thermal expansion of the magnetic material upon heating and its contraction during relaxation annealing (annealing out of free volume) or nanocrystallization (due to differences between specific mass the material in amorphous and nanocrystalline states). In addition, it has to allow application of external, either transversal or longitudinal (or both), magnetic field with respect to the orientation of the future magnetic flux lines of the device.

One possible example of furnace capable of transversal field annealing of ring-shaped magnetic circuits from rapidly quenched ribbons is a two-piece furnace with flat circular heaters from non-magnetic heating wires (Nicrothal). Ring-shaped core to be annealed is placed between the heaters containing a set of several thermocouples to control thermal gradient in the heated zone and the entire assembly is placed in a water-cooled gas-tight container in order to ensure a suitable annealing atmosphere (typically inert gas). The height of such assembly can be as low as 80 mm and can be placed between poles of a suitable electromagnet which can apply a field of up to 0.5 T. Figure 1 shows the realization of such device capable of annealing of ring-shaped with outer diameter up to 130 mm and height up to 25 mm.

Hysteresis loops of three 500 g ring-shaped cores wound from classical Fe-Cu-Nb-Si-B (Finemet) ribbons 25 mm wide in different applied transversal field are

shown in fig. 2. In all cases the cores exhibited coercive field < 0.9 A/m and total losses lower than 3 W/kg at 20 kHz and magnetization 0.3 T. This was achieved also due to successful control of the strain of individual turns of the ribbon on the underlying layers compensating the shrinkage of the ribbon imposed by nanocrystallization, however, without a significant decrease of the packing fraction of the core itself.



Fig. 1. Device for thermal annealing of ring-shaped cores in transversal magnetic field: 1, 2 – magnet polepieces, 3 – furnace with ring-shaped core

**Rys. 1.** Urządzenie do wyżarzania rdzeni pierścieniowych w poprzecznym polu magnetycznym: 1, 2 – jarzma magnetyczne, 3 – piec z rdzeniem pierścieniowym

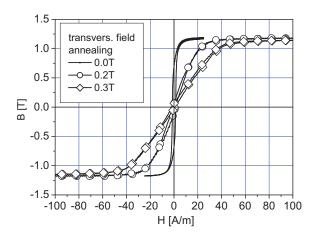


Fig. 2. Quasistatic, magnetic B(H) hysteresis loops of 500 g Finemet ring-shaped cores with outer diameter 130 mm annealed at 823 K for 1 hour without and with applied transversal magnetic field using the thermomagnetic annealing device

Rys. 2. Quasi-statyczne pętle histerezy magnetycznej B(H) rdzeni pierścieniowych o masie 500 g i średnicy zewnętrznej 130 mm, wyżarzonych w temperaturze 823 K przez 1 godzinę, w zróżnicowanym, poprzecznym polu magnetycznym

## 3. Testing of magnetoelastic properties of soft magnetic materials

Investigation on the magnetoelastic properties of soft magnetic materials is connected with measurements of magnetic hysteresis loop B(H) under the presence of mechanical stresses  $\sigma$ . On the result of these measurements parameters, important from technical point of view, can be calculated. As a result there are two most important requirements for magnetoelastic testing methodology:

- magnetic circuit of the sample has to be closed. If magnetic circuit is open, the significant values of demagnetization energy appear. As a result, permeability and stress sensitivity of such sample is significantly limited,
- distribution of stresses have to be uniform to enable physical interpretation of magnetoelastic phenomena.

Presented methods of magnetoelastic testing fulfill both these requirements for compressive and tensile stresses. As a result they are especially useful for magnetoelastic investigations.

#### 3.1. Frame shaped cores

In the case of bulk crystalline materials, such as steels or soft magnetic ferrites, the frame-shaped core can be used for magnetoelastic tests [12, 13]. The method of applying of the compressive force F to the frame-shaped sensing element is presented in fig. 3, whereas method of applying tensile stresses is presented in fig. 4.

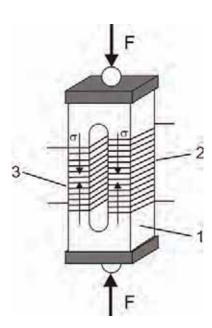


Fig. 3. Frame-shaped core subjected to compressive stresses: 1 – core under investigation, 2 – sense winding, 3 – magnetizing winding

Rys. 3. Rdzeń ramkowy w trakcie obciążania siłą ściskającą: 1 – rdzeń ramkowy, 2 – uzwojenie pomiarowe, 3 – uzwojenie magnesujące

The frame-shaped core provides the closed magnetic circuit. Due to the special nonmagnetic backings and force reversing mechanical system both the compressive and tensile stresses in the core's columns could be applied in the range up to  $100~\mathrm{MPa}$ .

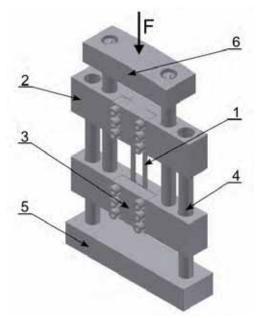


Fig. 4. Mechanical force F reversing system for application of the tensile stresses to the frame-shaped core: 1 – frameshaped core, 2 – moving element, 3 – sample holder, 4 – column, 5 – base, 6 – upper cover

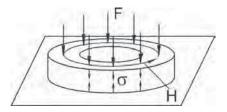
Rys. 4. Rewersor mechaniczny do zadawania naprężeń rozciągających do rdzenia ramkowego: 1 – rdzeń ramkowy, 2 – element ruchomy, 3 – uchwyt rdzenia, 4 – kolumna, 5 – podstawa, 6 – uchwyt górny

In the case of both tensile and compressive stress investigation both magnetizing and detecting winding were made on the frame-shaped core as it is presented in fig. 3. These winding enable measurements of the changes of the magnetic hysteresis loop B(H) under the influence of the stresses with standard histeresisgraph system.

#### 3.2. Ring shaped cores

In the case of ribbon magnetic materials, such as soft amorphous alloys, possibilities of application of frame-shaped cores for magnetoelastic tests are significantly limited. For this reason, in the case of these cores, ribbon ring shaped cores should be used. However, application of the force in direction of diameter of such core may lead to non-uniform distribution of stresses. Moreover in such a case, both compressive and tensile stresses are generated [14].

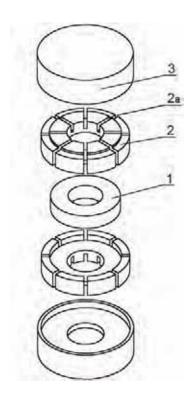
To achieve uniform distributions of stresses in ringshaped core, force should be applied perpendicularly to the base of the core as it is presented in fig. 5. It should be indicated, that this method creates the possibility of generation both compressive and tensile stresses in the core.



**Fig. 5.** Idea of the method of applying the uniform compressive stress to the ring core.

**Rys. 5.** Metoda zadawania jednorodnych naprężeń ściskających do rdzenia pierścieniowego

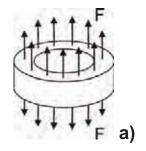
Device for generation of uniform, compressive stresses in the ring-shaped core [15] is presented in fig. 6. Base backings (3) allow a ring core (1) to be subjected of the compressive force F. Due to the special, nonmagnetic cylindrical backing (2) the distribution of stresses in the core is uniform. Measuring and magnetizing windings are placed in grooves (2a) at the cylindrical backings (2).



**Fig. 6.** Schematic diagram of the device for applying the uniform compressive stress to the ring core [12]: 1 – investigated ring core, 2 – nonmagnetic cylindrical backing, 2a – grooves for windings, 3 – base backings

Rys. 6. Urządzenie do zadawania naprężeń ściskających do rdzenia pierścieniowego: 1 – badany rdzeń, 2 – niemagnetyczne, cylindryczne nakładki, 2a – nacięcia na uzwojenie, 3 – nakładki bazowe

The idea of application of tensile stresses to the ring shaped core [16] is presented in fig. 7a, whereas technical device is presented in fig. 7b.



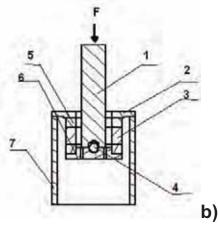


Fig. 7. Method of application of uniform tensile stresses to the ring-shaped sample: a) idea of the method, b) device for practical realization of this idea: 1 – main shaft, 2 – upper backing, 3 – ring-shaped core under investigation, 4 – steel ball, 5 – core backings, 6 – lower backing, 7 – tube

Rys. 7. Metoda zadania jednorodnych naprężeń rozciągających do rdzenia pierścieniowego: a) zasada działania, b) urządzenie do zadawania naprężeń rozciągających: 1 – wał główny, 2 – nakładki górne, 3 – badany rdzeń pierścieniowy, 4 – kulka stalowa, 5 – nakładki rdzenia, 6 – nakładki dolne, 7 – rura

Ring-shaped core (3), subjected to magnetoelastic tests, is fixed to core backings (5). Next, each of these backings was fixed to upper backing (2) or lower backing (6). In backings (5) and (6) special holes were drilled, to enable core to be winded by magnetizing and sensing windings. Compressive force F generated by hydraulic press is transferred by shaft (1) and ball (2) to the lower backing (6). Presented device acts as mechanical reversing system. As a result, compressive force F generates uniform tensile stresses in the core (4). Also in this case, the changes of magnetic hysteresis loop B(H) under the influence of the stresses are measured with standard hysteresis graph system.

Example of such result achieved for Fe<sub>77</sub>Cr<sub>2</sub>B<sub>16</sub>Si<sub>5</sub> in as-quenched state is presented in fig. 8. In spite of the fact, that magnetoelastic effects for compressive and tensile stresses were measured with different mechanical setups,  $B(\sigma)_H$  dependences are continuous. Moreover maximum on the  $B(\sigma)_H$  characteristics can be observed. This is so called Villari point [17], which is very important for theoretical explanation of magnetoelastic Villari effect.

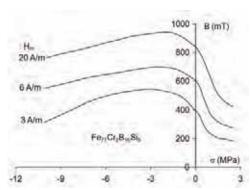


Fig. 8. Magnetoelastic  $B(\sigma)_H$  characteristics of amorphous alloy in as-quenched state for constants values of amplitude of magnetizing field  $H_m$ 

**Rys. 8.** Charakterystyki magnetosprężyste  $B(\sigma)_H$  stopu amorficznego o składzie  $Fe_{77}Cr_2B_{16}Si_5$  w stanie wyjściowym uzyskiwane dla ustalonych wartości amplitudy natężenia pola magnesującego  $H_m$ 

#### 4. Summary

Methodology of thermal annealing presented in the paper together with methods of testing the magnetoelastic properties of soft magnetic materials create new possibility of testing the functional properties of these materials for industrial applications. Utilizing these methods, not only magnetic, but also magnetoelastic properties of magnetic materials may be optimized. It is especially important in the case of the modern, miniaturized components, where even small forces may lead to significant stresses. This may result in changes of functional properties of the core of inductive component leading to malfunction of electronic device, such as switching mode power supply.

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#### Wyżarzanie materiałów magnetycznie miękkich i metody badania ich właściwości magnetosprężystych

Streszczenie: W artykule przedstawiono zarówno nową metodę relaksacji termicznej w materiałach magnetycznie miękkich, jak i metody pomiaru charakterystyk magnetosprężystych w tych magnetykach. W artykule przedstawiono także wybrane wyniki pomiaru wpływu procesu relaksacji termicznej w obecności pola magnetycznego na charakterystyki magnesowania stopów amorficznych, jak również wyniki pomiaru charakterystyk magnetosprężystych. Należy podkreślić, że z wykorzystaniem przedstawionej w pracy metodyki możliwy jest pomiar charakterystyk magnetomechanicznych zarówno w zakresie naprężeń ściskających, jak i rozciągających.

**Słowa kluczowe:** materiały magnetycznie miękkie, wyżarzanie, właściwości magnetosprężyste

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