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# Properties changes of Co-based amorphous alloy in thermal activation process

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## ABSTRACT

**Purpose:** The paper presents a stress relaxation process of Co69Mo2Fe4Si14B11 metallic glass and properties changes in a temperature range up to 300 ºC after annealing from 1 to 16 hours (in case of plasticity and stress relaxation researches) and 2 hours (for the magnetic properties investigations).

**Design/methodology/approach:** The plastic properties of the studied material were determined using the bend test, applied for metallic glasses. The investigations of magnetic properties were carried out using the Ferrometer measurement system. For the higher frequencies the Remacomp C-100 system was used, allowing determination of the magnetic properties of the ferromagnetic cores. Stress relaxation investigations in metallic glasses by a bending test were carried out.

**Findings:** The influence of thermal activation on the properties changes of Co-based metallic glass was determined after annealing from temperature of 150 to 300°C. After the heat treatment carried out the best magnetic properties were obtained for the sample annealed for 2 hours at 150°C. The increase of the magnetic field frequencies causes a significant deterioration of the material magnetic properties. High plasticity the metallic glass ribbons have to 200°C during annealing to 16 hours. Increasing the annealing temperature causes brittleness of the material. Distinct relaxation processes are observed at higher temperatures and annealing times.

**Research limitations/implications:** Metallic glasses after heat treatment become brittle, which reduces the possibility of using these materials, and often impossible to change their geometric form. Therefore, the heat treatment of metallic glasses must be carried out after the final forming of the geometry of shaped, cores, etc., or in conditions not leading to embrittlement of the material.

**Practical implications:** Usage of metallic glasses is possible only in a narrow range of temperatures which does not lead to significant changes of properties or after proper heat treatment carried out in the aim of specified physical properties obtaining. The important is prediction of alloy properties changes during temperature changes and material using.

**Originality/value:** In the article influence of low-temperature thermal activation processes, which was conducted up to 16 hours, on the stress relaxation and significant changes of magnetic properties of cobalt-based metallic glass were presented.

**Keywords:** Amorphous Materials; Thermal activation; Relaxation; Magnetic properties

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MATERIALS

## 1. Introduction

Most existing engineering materials, especially those used in structural applications, are characterized by a crystalline structure. However, since produced a material having an amorphous structure, numerous studies on the properties and possibilities of its application also as structural materials are carried out. Among these materials particularly important are metallic glasses. These materials are formed through the very rapid cooling of the liquid metal, so that the crystallization process is skipped, and the material assumes an amorphous structure [1-3].

The properties of metallic glasses dependent on the chemical composition. As a result of numerous studies carried out the metallic glasses shown that these materials are characterized by a high corrosion resistance, high strength, and good deformability at the same time they do not exhibit brittleness [4-7].

Two types of structural changes occurring in these materials have impact on the physical, mechanical and chemical properties of metallic glasses. There are [8-12]:

a) structural relaxation phenomenon in amorphous phase,
b) crystallization of amorphous phase by diffusive phase transformation.

The structural relaxation is observed already at room temperature and even at lower temperatures. Relaxation is accompanied by a changing of physical properties of the material and the stage is sometimes partially reversible. At higher temperatures the dominant process is irreversible crystallization passes through the nucleation and diffusive growth of crystals [13-18].

Stress relaxation studies show that during annealing at the appropriate temperature comes to the processes causing significant reduction of internal stress, which also confirms that the structural relaxation is proceed in material. It leads to changes in the atomic configuration in the areas of short range ordering - mainly in the topological nature of amorphous structure [8,9,14,19,20].

The structural relaxation is also associated with diffusion, which involves the rearrangements of individual atoms from defect to defect, even on multi-atomic size. It is related closely with the viscosity and is responsible for relaxation of mechanical stresses and strains. This process can occur at low temperatures (about 150°C) [9,10,12].

Metallic glasses are characterized by rather good plasticity and, most importantly, the lack of consolidation. However, increase of the temperature during annealing causes decrease of the metallic glasses plasticity until complete it disappearance at the time of crystallization [21-23].

Interesting properties show magnetically soft materials made from ferromagnetic metallic glasses. These materials have quite large saturation magnetization $B_S$ when there is a low coercive force $H_c$, which are achieved in the external magnetic field about low intensity. They are characterized by a high magnetic permeability μ. Soft magnetic metallic glasses also have a small anisotropy, and a high Curie temperature [22, 24-28].

Metallic glasses connecting the properties of conventional crystalline materials with a unique amorphous structure are becoming very good substitutes in many applications. For example, the ferromagnetic metallic glass used in the manufacture of transformer cores, but also in production of magnetic screens which are designed to protect against electromagnetic, electric and magnetic fields [29].

Metallic glasses are inherently unstable and susceptible to crystallization under the influence of thermal activation [30-35]. This make their use in conditions leading to significant changes of properties during exploitation impossible.

The usage of metallic glasses depends, therefore, on the possibility of predicting and controlling of the alloy properties during temperature changes in operation or control of properties of amorphous alloys by appropriate heat treatment [36].

## 2. Research methodology

### 2.2. Material for reseach

The multicomponent alloy with amorphous structure in a thin ribbon form was produced by continuous casting of the alloy stream on the surface of the vibrant roll. The investigations were conducted on the metallic glass ribbons about the chemical atomic composition: $Co_{69}Mo_2Fe_4Si_{14}B_{11}$, 0.028 mm thickness and 10 mm width.

### 2.2. Heat treatment

The process of low-temperature heat treatment consisted in annealing of metallic glasses in an electric chamber furnace Thermolyne 6000 type with electronic temperature controller, in which the accuracy of the temperature measurement and control is within ± 2°C. The ribbons were annealed at the 150, 200, 250 and 300°C. The annealing time was in the range of 1 to 16 hours (in case of plasticity and stress relaxation researches).

The cores for magnetic properties investigations annealed at temperature 100 to 300°C with 50°C gradation, keeping the annealing time 2 hour.

In order to protect the samples against possible oxidation, samples were additionally by tight protection of aluminum foil protected.

### 2.3. The magnetic properties investigations

The magnetic properties investigations were carried out for the ring cores from the $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ metallic glass ribbons.

The cores weighing about 10 grams had an internal diameter 31 mm and external 35 mm. The cores are wound as a compact ribbon coils with a width of the core equal to the width of the ribbon. The investigations were carried out using the Ferrometer measurement system. For the higher frequencies the Remacomp C-100 system was used, what allows to determination of the magnetic properties of the ferromagnetic core.

The cores were placed in a polyamide carcass with internal diameter 31 mm and external diameter 35 mm, in which then wound 80 primary and 80 secondary coils.

To carried out an investigation of the magnetic properties were determined following initial values:

$L_{av}$ - average length of magnetic path:

$$L_{av} = \Pi \cdot d_{av} \tag{1}$$

$d_{av}$ - average diameter expressed by the formula:

$$d_{av} = \frac{d_e + d_i}{2} \tag{2}$$

where:
$d_z$ - outside diameter of the ring,
$d_w$ - inside diameter of the ring.

The formula for the cross-sectional area S represents the relationship:

$$S = \frac{M}{L_{av} \cdot \rho} \tag{3}$$

where:
M - mass of the investigated sample,
ρ- material density ($\rho = 7.8$ g/cm$^3$).

## 2.4. The plasticity investigations

The samples of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ metallic glass for plasticity investigations were prepared in the ribbon sections form about 100 mm length. The plasticity investigations were carried out on five samples for each state of the tested materials, then the average value was determined.

The plastic properties of the studied material were determined using the bend test, applied for metallic glasses. In this method the sample was bent in measuring holders up to durable rise of maximum deformation or to a crack creation. The plasticity deformation answering the yield stress was calculated from the expression:

$$\varepsilon = \frac{g}{D - g} \tag{4}$$

where:
g - ribbon thickness,
D - distance between jaws at which plastic durable deformation follows or fracture.

When the sample despite the closure of the jaws to the end will not be destroyed, the plastic deformation is as follows:

$$\varepsilon = \frac{g}{2g - g} = 1 \tag{5}$$

This means that the sample has a very good plasticity and then assumes a value equal 1. In other cases, the plasticity assumes a value between 0 and 1.

## 2.5. Stress relaxation investigations

The process of relaxation investigations involves annealing ribbons with a set value of initial stress. The ribbons are rolled up in a circle with a given diameter and inserted into the collet (Fig. 1).

The samples for the stress relaxation investigations was made in the ribbon sections form with 50 mm length, which were coiled in a ring and deposited on the cylinder with 10 mm external diameter, clenching them from the outside by the ring about internal diameter 10.15 mm.

Fig. 1. The collet used for stress relaxation investigations

After stabilization of the ring in the outer constrains, the sample was annealed in an appropriate temperature - time conditions, then cooled to room temperature and removed external constraints.

During heat treatment, the stress $\sigma_o$ after the relaxation time τ is changed to the value of $\sigma_t$. As a result of the relaxation reduction the part of introduced stress, the sample of ribbon after the loosing of external constraints does not return to its original form showing the curvature of radius (Fig. 2).

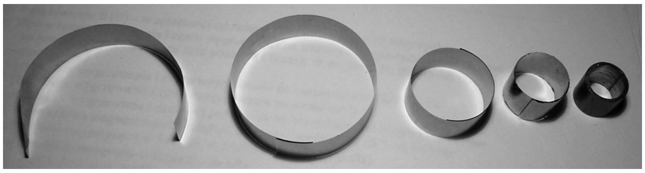

Fig. 2. Exemplary view of relaxed ribbon after losing inner constraints

The diameters of the ribbon is measured directly or indirectly determined by measuring the length of the chord and distance of this chord from the perimeter of the ribbon as shown in Fig. 3.

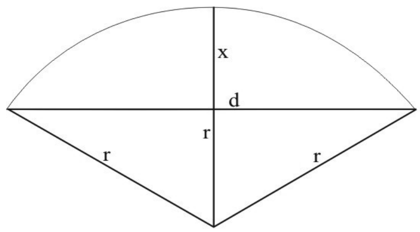


Fig. 3. The method of determining the diameter of a circle, having data: di the chord length d and distance of this chord from the perimeter x

Having d and x we can determined diameters of the ribbon after expanding by calculating the radius r from the formula:

$$r^2 = (\frac{d}{2})^2 + (r-x)^2 \quad (6)$$

As a result of determining of the diameter value d = 2r, is possible to calculate the coefficient of stress relaxation (stress loss) from dependence:

$$\eta = \frac{d_o}{d_t} \quad (7)$$

where:
$d_o$ - inside diameter of the coil ribbon during heat treatment,
$d_t$ - diameter of the coil ribbon after annealing and release of outer constraints.

## 3. Results and discussion

### 3.1. Magnetic properties investigation results

As a result of magnetic properties investigations for particular samples of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ metallic glass in 'as quenched' state and after heat treatment performed the hysteresis loop was obtained. From the hysteresis loop the coercive force $H_c$, residual magnetism (remanence) $B_r$, maximum induction $B_{max}$, magnetic field strength $H_{max}$ were read. The initial magnetic permeability $\mu_{start}$ and changes of permeability as a function of field strength $\mu_{max}$ were determined.

The best magnetic properties of the material occur at the largest values of $B_r$, $B_{max}$ and μ, and the smallest value of the coercive force $H_c$, that is at the most narrow and a high hysteresis loop.

The $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ alloy in the 'as quenched' state is characterized by narrow hysteresis loop, typical of soft magnetic materials (Fig. 4). It has good magnetic properties ($H_c$ = 1.95 A/m, $B_r$ = 0.38 T, $B_{max}$ = 0.625 T). This alloy also characterizes a high magnetic permeability about the value $\mu_{max}$ = 188237.

After annealing at 150 to 300°C for 2 h the studied alloy has |a rectangular hysteresis loops with coercive force in the range of 1.79 to 3.08 A/m and values of magnetic induction 0.66 to 0.72 T.

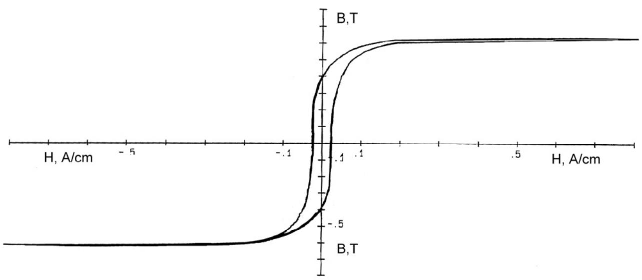


Fig. 4. Hysteresis loop of Co69Mo2Fe4Si14B11 alloy in as quenched' state

In comparison with 'as quenched' state, the samples annealed at 150 and 300°C for 2 hours have lower coercive force, equal 1.79 A/m and 1.92 A/m, what is an advantageous phenomenon.

The other samples annealed for 2 hours at 200°C and 250°C have a larger coercive force. However, the sample annealed at 200°C has an adversely high value of coercive force $H_c$ = 3.08 A/m.

The maximum initial magnetic permeability $\mu_{start}$ = 79780 was also found for the sample annealed at 150°C. The highest value of magnetic permeability $\mu_{max}$ = 168396 obtained for the sample annealed at 300°C. However, the sample annealed at 250 and 300°C are in a brittle state. Therefore, from the samples characterized by a plastic state, i.e. for samples annealed at 150 and 200°C the highest value of magnetic permeability $\mu_{max}$ = 148756 has a sample annealed at 150°C.

The results of characteristic magnetic properties of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$alloy in 'as quenched' state and after heat treatment are presented in Table 1 and Fig. 5.

Table 1.
Magnetic properties of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ metallic glass after heat treatment for 2 h

| Material state | $H_c$ A/m | $H_{max}$ A/m | $B_r$ T | $B_{max}$ T | $\mu_{start}$ | $\mu_{max}$ |
|---|---|---|---|---|---|---|
| 'as quenched' state | 1.95 | 78.9 | 0.38 | 0.625 | 73627 | 188237 |
| 150°C | 1.79 | 71.4 | 0.39 | 0.683 | 79780 | 148756 |
| 200°C | 3.08 | 87.3 | 0.39 | 0.722 | 56757 | 90031 |
| 250°C | 2.57 | 80.9 | 0.34 | 0.657 | 67140 | 92274 |
| 300°C | 1.92 | 84.5 | 0.54 | 0.665 | 75825 | 168396 |

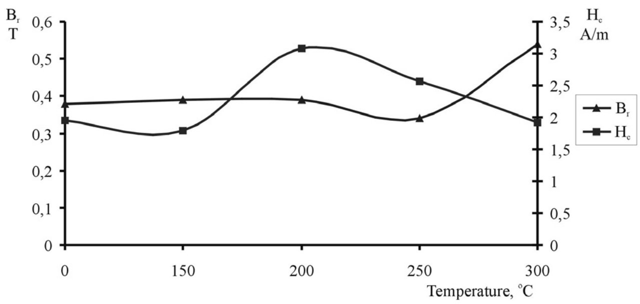


Fig. 5. Influence of annealing temperature on the remanence $B_r$ and coercive force $H_c$ of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ alloy

Influence of annealing temperature on the initial and maximum magnetic permeability are shown in Figs. 6, 7.

The best magnetic properties of the studied $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ metallic glass have samples annealed at 150°C retaining in these temperature - time conditions high plasticity. For a sample in this state and in the 'as quenched' state additionally investigations of magnetic properties in the higher frequency range (up to 300 kHz) on Remacomp device were carried out.

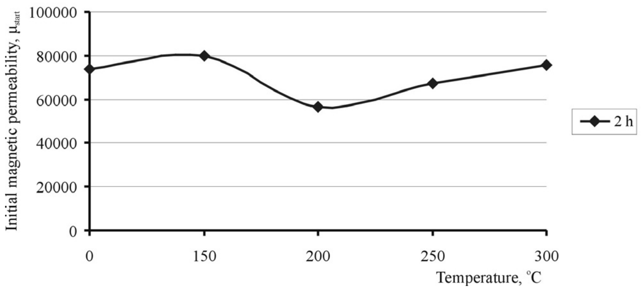


Fig. 6. Influence of annealing temperature on the initial magnetic permeability $\mu_{start}$ of the studied metallic glass

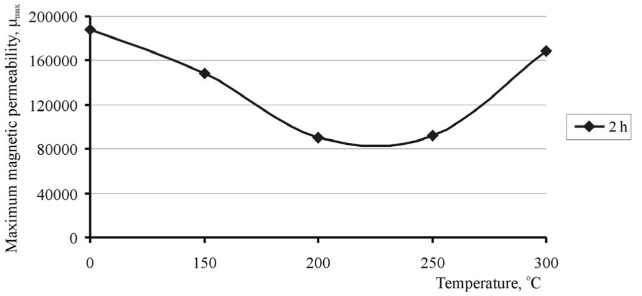


Fig. 7. Influence of annealing temperature on the maximum magnetic permeability $\mu_{max}$ of the investigated material

Table 2.
Magnetic properties of metallic glass ribbon after annealing at 150°C at a constant maximum induction $B_{max}$ = 0.4 T, at frequencies in the range up to 300 kHz

| f kHz | $H_c$ A/m | $H_{max}$ A/m | $B_r$ T | $B_{max}$ T |
|---|---|---|---|---|
| 20* | 12.8 | 79 | 53.7 | 61 |
| 50 | 15.6 | 16.9 | 0.344 | 0.4 |
| 100 | 21 | 23.5 | 0.365 | 0.4 |
| 300 | 38.3 | 43.7 | 0.388 | 0.4 |

* for 20 kHz the investigations in 'as quenched' state were carried out

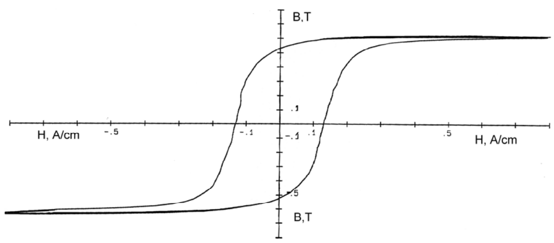


Fig. 8. Hysteresis loops obtained for frequencies 20 kHz for cores from $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ alloy in 'as quenched' state

With increasing frequency of magnetic field the hysteresis loops extend, and therefore significantly increased the coercive force (from $H_c$ = 15.6 A/m at f = 50 kHz and $H_c$ = 38.3 A/m at f = 300 kHz). This was accompanied by a slight increase in remanence $B_r$ (0.344-0.388 T).

The results of magnetic properties investigations of the samples in 'as quenched' state at 20 kHz frequency and after annealing at 150°C at frequency range up to 300 kHz are shown in Table 2 and Figs. 8, 9.

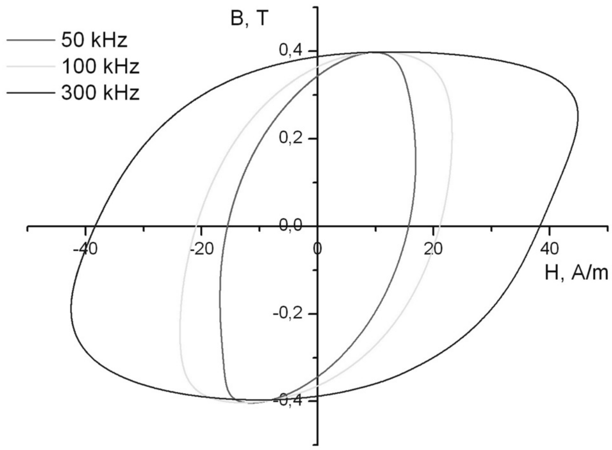


Fig. 9. Hysteresis loops obtained for frequencies in the range of 50-300 kHz for cores from $Co_{69}Mo_2Fe_4Si_{14}B_{11}$alloy annealed at 150°C

## 3.2. Plasticity investigation results

Based on the obtained results, it was found that samples of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ metallic glass retained high plasticity (ε = 1) at 150 and 200°C for an annealing time to 16 hours. For these cases, none of the investigated ribbon sections has not fracture during the bend test.

The ribbons annealed at 250 and 300°C showed loss of a high plasticity after 1 hour. The samples brittle cracked during bending.

When annealing time elongated the plasticity of the material was significantly reduced.

Specification of plasticity changes results shown in Table 3.

Dependence of average plastic strain ε on time and temperature annealing are shown in Fig. 10.

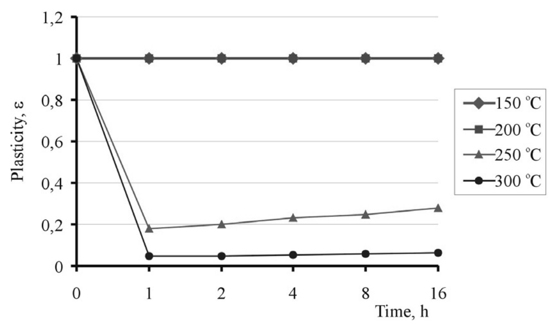


Fig. 10. Dependence of plasticity ε on annealing time of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$metallic glass at various temperature

Table 3.
Influence of time and temperature annealing on the plasticity of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$metallic glass

| Temperature | Time | | | | |
|---|---|---|---|---|---|
| | 1 h | 2 h | 4 h | 8 h | 16 h |
| 150ºC | 1 | 1 | 1 | 1 | 1 |
| 200ºC | 1 | 1 | 1 | 1 | 1 |
| 250ºC | 0.178 | 0.20 | 0.23 | 0.248 | 0.28 |
| 300ºC | 0.045 | 0.048 | 0.053 | 0.058 | 0.062 |

### 3.3. Stress relaxation investigation results

Stress relaxation investigations showed that the metallic glass ribbons annealed at 150°C in time to 16 hours did not undergo stress relaxation process or their size is very small - practically unmeasurable.

This is due to the fact that the material after removal of the external constraints did not show visible deformation. Stress relaxation coefficient with increasing temperature and annealing time increases its value. The deformation of relaxation achieved a lowest measurable value equal $\eta \approx 0.11$ for sample annealed at 200°C for 1 hour. The largest deformation, $\eta \approx 0.82$ has metallic glass ribbon annealed in the longest time and at highest temperature (16 hours, 300°C).

The results of stress relaxation of metallic glass ribbons are presented in Table 4 and shown in Fig. 11.

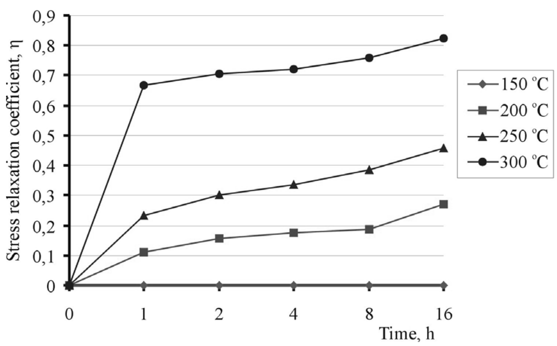


Fig. 11. Dependence of stress relaxation coefficient η on annealing time at different temperatures

Table 4.
Influence of time and annealing temperature on the coefficient of $Co_{69}Mo_2Fe_4Si_{14}B_{11}$metallic glass relaxation η

| Temperature | Time | | | | |
|---|---|---|---|---|---|
| | 1 h | 2 h | 4 h | 8 h | 16 h |
| 150°C | very low ~0 | very low ~0 | very low ~0 | very low ~0 | very low ~0 |
| 200°C | 0.112 | 0.156 | 0.176 | 0.185 | 0.271 |
| 250°C | 0.232 | 0.303 | 0.335 | 0.385 | 0.457 |
| 300°C | 0.669 | 0.706 | 0.721 | 0.757 | 0.825 |

## 4. Conclusions

The magnetic properties of metallic glass ribbons produced from $Co_{69}Mo_2Fe_4Si_{14}B_{11}$ alloy after heat treatment in the 150 - 300°C temperature range with 50°C gradation, during two hours are changing.

In the ‘as quenched’ state studied material has good magnetic properties (narrow hysteresis loop, high remanence, low coercive force and high magnetic permeability).

From among samples annealed for 2 hours the best magnetic properties has the sample annealed at 150°C (the smallest coercive force $H_c$ equal 1.79 A/m and maximum initial magnetic permeability $\mu_{start}$ = 79780). For these heat treatment conditions the measurements of magnetic properties at higher frequencies also carried out. A significant deterioration of the material magnetic properties at frequencies of 50, 100 and 300 kHz were observed. First of all coercive force has increased, what is a negative phenomenon. At the values of coercive force $H_c$ and the magnetic permeability μ of the magnetic materials have influence various factors, in this case a significant are the advancement of structural relaxation processes and stress relaxation. The purpose of heat treatment is improvement of the material magnetic properties in comparison with ‘as quenched’ state. The width of hysteresis loop is highly dependent on frequency. As frequency increases, the hysteresis loop is expanding, what is associated with decrease of material magnetic properties. This is mainly connected with dynamic effects relating to remagnetization of magnetic domains.

The temperature and annealing time also have a significant impact on the plasticity changes of the investigated material.

The Co-based metallic glass ribbons have high plasticity $\varepsilon = 1$ to 200°C in annealing time to 16 hours. Increase of annealing temperature causes embrittlement of the material after annealing in 1 hour. Elongation of annealing time causes a further increase of the brittleness.

At the same time the loss of plasticity was reduced to internal stresses as a result of advanced relaxation processes.

Stress relaxation investigations have shown that in initial stage the stress relaxation coefficient is very small or equal zero.

The measurable effects of relaxation are only after annealing at 200°C in 1 hour. In this conditions the ribbon, after release of constraints is not returned to its original form.

The explicit relaxation processes are noticeable at higher temperatures and annealing times.

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