

Structure and properties changes of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ metallic glass by low-temperature thermal activation process

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Materials

ABSTRACT

Purpose: The paper presents a structural relaxation process of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ metallic glasses and structure and properties changes in a temperature range up to 300°C after annealing from 2 to 16 hours.

Design/methodology/approach: The relaxation and crystallization of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ metallic glasses were examined by mechanical test, relaxation test, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) methods.

Findings: The influence of thermal activation on the structural relaxation process of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ metallic glasses was determined after annealing from temperature of 100 to 300°C. The beginning of the structural relaxation was revealed after annealing at 100 and 150°C, especially after long annealing times of 8 and 16 hours. The structural relaxation process was confirmed by examination of dimensional changes of samples associated with partial reduction of free volume and the ordering of topological and chemical structure of metallic glass. Significant changes in the structure and properties of the alloy was observed after annealing at 300°C. The reduction of tensile strength and high fragility of samples was also determined. This decrease is associated with extending of the structural relaxation and beginning of crystallization process by formation of small crystallites of α -Fe phase in amorphous matrix.

Research limitations/implications: The structural relaxation process and beginning of crystallization on changes of strength, ductility, fracture morphology, structure, process of stress relaxation and geometry of studied alloy were also achieved in function of temperature and time of annealing.

Practical implications: The course of relaxation processes can be used for analysis of thermal stability of metallic glasses.

Originality/value: The paper presents a significant influence of low-temperature thermal activation, which was conducted up to 16 hours, on the structural relaxation and changes of selected mechanical properties.

Keywords: Amorphous materials; Relaxation; Mechanical properties; Fracture morphology

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1. Introduction

Metallic glasses are achieved from alloys with a specific chemical compositions, which allow to obtain amorphous materials by casting of liquid alloy with applied high cooling rates. Such materials are characterized by non-crystalline structure and exhibit an unconventional physical and chemical properties. Metallic glasses are produced on an industrial scale and they have found many applications in modern energy-efficient applications and construction solutions. Special interest of metallic glasses was found in their good magnetic, mechanical and electrical properties [1-4].

Metallic glasses have metastable structure. Thermally activated processes can change their properties due to structural phenomena leading to the final stage of crystallization and the existing of structures corresponding to a thermodynamic equilibrium. Thermal activation of metallic glasses can be conducted to forming their physical properties with the amorphous structure [5-8].

Knowledge of the effect of thermal activation of metallic glasses on the changes of physical properties also allows to predict areas of their practical application. In some cases, these changes caused a reduction of the desired properties of metallic glasses and preventing their using. Many studies of amorphous alloys have omitted very important influence of heat treatment on forming materials properties. They have determined the dependence of structural changes and properties of metallic glasses as a function of annealing for constant heating rate or annealing in a relatively short time. The practical using of metallic glasses depends on the ability to predict and control their thermal stability. The forming of metallic glasses properties during temperature changes is also related with influence of temperatures during their using or control by suitable heat treatment [5-15].

Two kinds of structural changes are occurred in metallic glasses, which influence on physical, mechanical and chemical properties of these materials. These are [5-10, 15-19]:

- 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. It occurs to begin the crystallization temperature T_x or the glass transition temperature T_g , when $T_g < T_x$. The relaxation is accompanied by a changing of physical properties of the material. That stage is sometimes partially reversible. At higher temperatures the dominant is irreversible crystallization process, which proceed by the nucleation and diffusive growth of crystals. The theory of structural relaxation phenomena in metallic glasses consists of two groups of issues. These problems are relating to the atomic structure and defects of glassy alloys, the changes in the structure during the structural relaxation and the theory of the kinetics of these changes [8].

The structural relaxation can be divided into two processes (rearrangements of atoms in the amorphous phase) [10]:

- a) topological short-range ordering – TSRO,
- b) chemical short range-ordering – CSRO.

These phenomena are actually the main atomic processes taken into account in determining the kinetics of the structural relaxation. Components of TSRO and CSRO are associated with

rearrangements of atoms (or groups of atoms) at short range. These rearrangements are resulted by changing of various types of displacement of atoms or improving their distribution in the sample volume [8].

In the initial stage of the structural relaxation with the CSRO ordering, there are forming the pair of atoms. In the case of suddenly changes of temperature (T) their concentration is also changed. The CSRO process is therefore related to rearrangement of given type of atoms. The degree of atomic ordering depends reversibly on the temperature. TSRO process is associated with the annihilation of pairs of defects (n-p), redistribution and the reduction of free volume (microvoids) as an irreversible process [8, 20, 21].

Stress relaxation studies inform that during annealing at the appropriate temperature in material has been done a significant reduction of internal stress. The reduction of stress also confirms that the structural relaxation is existed in material and leads to changes in the atomic configuration in the areas of short range ordering [8, 10, 19].

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 it is responsible for relaxation of mechanical stresses and strains. This process can occur at low temperatures (about 150°C) [18, 19].

The term of a free volume applies to the additional volume frozen as a result of casting processes, such as during rapid cooling from the liquid. This volume does not contain the volume of local defects, which also contains a sample fully relaxed at a given temperature. It should also be noted that in the amorphous sample are local areas of smaller voids that occur due to different sizes of atoms forming the glassy alloy. While frozen free volume usually extends over a distance several atoms, whereas the volume associated with structural defects by displacements of individual atoms. The free volume changes are involved in irreversible component of TSRO and reversible changes in CSRO [8, 10, 19].

After the structural relaxation, the alloy has an amorphous structure, but after the partial or complete crystallization the material exhibits a mixed structure with amorphous and crystalline phase. Therefore, samples with that structure are no longer to use in practice in the form of ribbons due to their significant fragility [22].

The presented results are a continuation of studies of the structure and physical properties of metallic glasses by thermally activated processes during structural relaxation and crystallization behavior [7, 23-25].

2. Material and research methodology

The influence of low-temperature heat treatment in range of 100, 150, 200, 250 and 300°C for annealing times of 2, 4, 8 and 16 hours was examined to study the changes of dimensions of samples, mechanical properties, stress relaxation phenomena, material structure and fracture morphology. As starting materials for tests were used metallic glasses in as-cast state.

The study was conducted on the metallic glass ribbons with chemical composition equivalent to the following atomic shares:

$\text{Fe}_{78}\text{Si}_9\text{B}_{13}$. Tested ribbons had a thickness of 0.028 mm and width of 9.60 mm. The edges of the samples did not show defects from casting process because the tested material was cut from the ribbons of greater width (Metglas 2605, Allied Signal).

The tensile tests were conducted on Instron type 1295 machine. The tensile strength was calculated from the following dependence:

$$R_m = F_m / S_0 \quad (1)$$

where:

F_m – tensile force (maximal),

S_0 – cross section of sample.

The number of tests was carried out by 10 samples for each state. The samples of ribbon sections about 120 mm were fixed in flat holder with rubber facing. Distance among grips of tensile machine was 50 mm and the speed of machine beam was 5 mm per minute.

The plastic proprieties 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 propagation. The plasticity deformation answering the yield stress was calculated from the expression:

$$\varepsilon = g / (D - g) \quad (2)$$

where:

D – distance between jaws at which plastic durable deformation follows or fracture,

g – ribbon thickness.

Stress relaxation investigations were done by bend test. The studied segments of ribbons with 60 mm length were coiled in rings around a cylinder of diameter of $d_0 = 10$ mm, which led to the introduction of selected elastic stress σ_0 . After stabilization of the ring in the outer constraints, the sample was annealed in an appropriate temperature and time conditions. Then, it was cooled to room temperature and external constraints were removed (Fig. 1).

During heat treatment the stress σ_0 after the relaxation time τ is changed to the value of σ_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 with radius of r_t ($d_t = 2r_t$). The relaxation process of collapsed in the ring material can be

described by the equation $\sigma_t = \sigma_0 (1 - d_0/d_t)$. Coefficient of stress relaxation (stress loss) during the annealing is $\eta = d_0/d_t$.

The study of sample density changes were estimated indirectly by measuring the length changes of the samples. Specimens of initial length of about 100 mm for each heat treatment condition were tested.

In order to accurately determine the length of the sample, ends of ribbons on both sides were cut down at an angle of 45° and accurately measured by the measuring microscope with an accuracy of 0.01 mm.

The samples were annealed and cooled in air. After heat treatment the samples were measured again precisely to determine the change of length under the influence of annealing and determined the percentage change in the length of the studied samples. Absolute measurement error was 0.001%.

The tensile fracture surface was examined by „Opton” type DSM 940 scanning electron microscopy. For that examination the secondary electrons emission was used at 20 kV voltage and magnification from 1000 to 3500 times. The investigations of structure were performed on thin foils by the method of transmission electron microscopy (Tesla BS 540).

3. Results and discussion

The studied alloy in as-cast state has an amorphous structure. It has high tensile strength of 1740 MPa and the rate of high plasticity ($\varepsilon = 1$). Research of fracture morphology achieved in a tensile test showed that it has ductile fracture with “river” patterns, which is characteristic for metallic glasses with high strength and ductility (Fig. 2).

The structure of thin foils observed by TEM methods shows a characteristic contrast of the amorphous structure, which revealed no coherent scattering reflections. Electron diffraction of such structure exhibits a broad diffraction pattern typical for an amorphous structure (Fig. 3).

Annealing of the studied alloy at 100 and 150°C at the time up to 16 hours does not lead to significant changes in tensile strength ($R_m \cong 1720$ to 1680 MPa with a standard deviation $\sigma \cong 130$ MPa). The high plasticity of samples is achieved ($\varepsilon = 1$). The fracture morphology of the samples after strength tests is similar to that in the initial condition for short annealing times (2 hours).

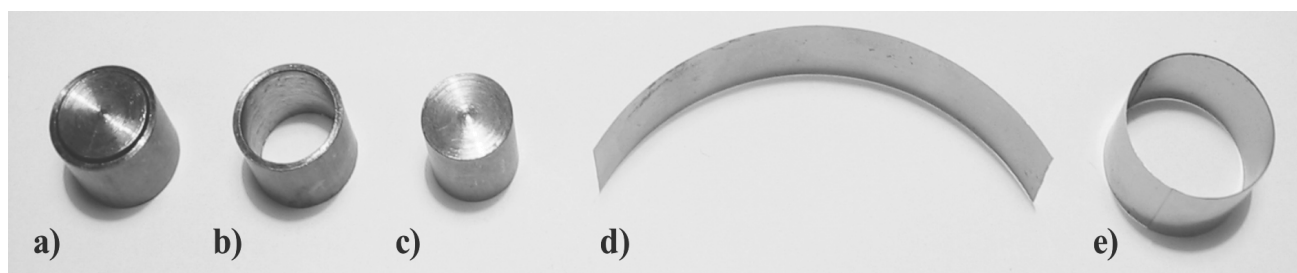


Fig. 1. Investigations of stress relaxation in metallic glasses by a bending test: a) sample for relaxation test: internal cylinder – coiled ribbon – outer ring for fixing ribbon, b) outer ring, c) internal cylinder for coiled ribbon, d, e) exemplary view of relaxed ribbon after loosing inner constraints

The increasing of annealing time caused a formation of diverse areas with a greater density of minor “veins” (Fig. 4). That result may inform of the beginning of structural relaxation process. Structure investigations of thin films (TEM) show the presence of the amorphous structure.

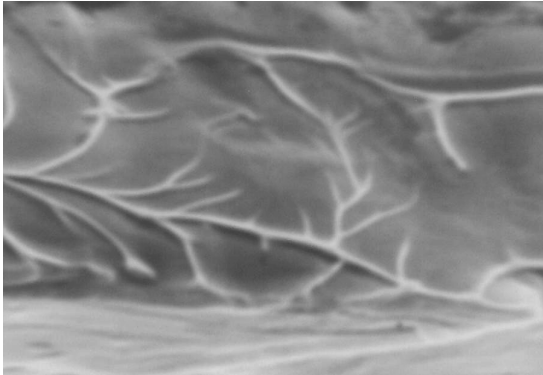


Fig. 2. Fracture morphology of strength sample of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy in as-cast state (ductile and “river” fracture, SEM, magn. 4 000x)

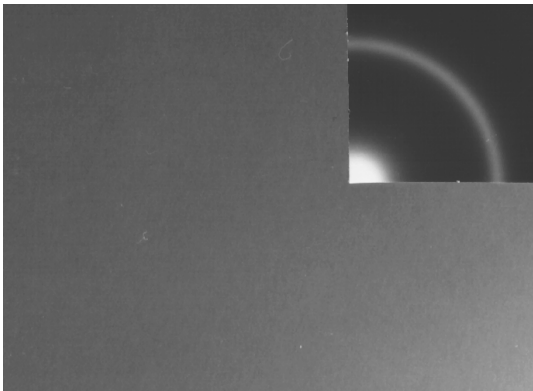


Fig. 3. Transmission electron micrograph and electron diffraction pattern of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy in as-cast state (TEM, magn. 60 000x)

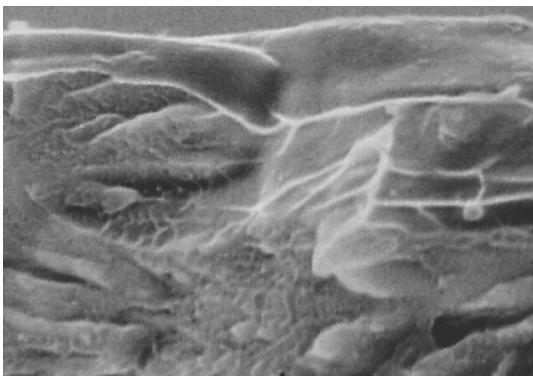


Fig. 4. Fracture morphology of strength sample after annealing of 100C/8h of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy (mixed fracture with “river” matrix, SEM, magn. 3 000x)

A process of structural relaxation initiated at 100 and 150°C was confirmed by studies of dimensional changes of samples and tests of stress relaxation processes. In range of the dimensional changes of samples, the reducing of the length can be observed on a scale larger than the measurement error after annealing at 100°C at time over than 8 hours (Fig. 5). Stress relaxation tests at this temperature allowed the observation of small changes after 16 hours (Fig. 6).

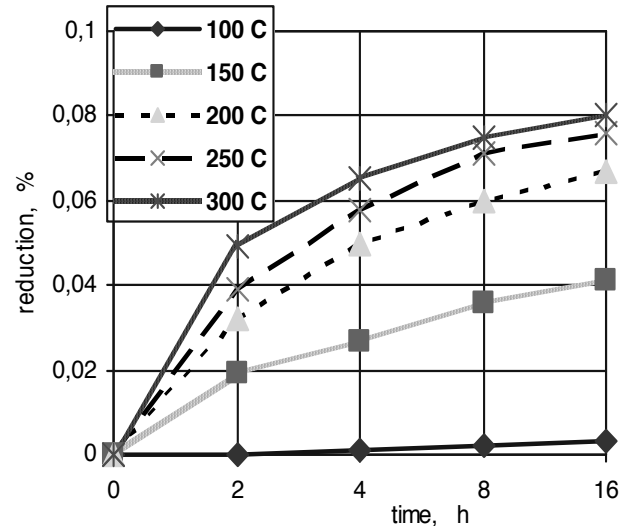


Fig. 5. Influence of temperature and time of annealing on length changes of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy samples

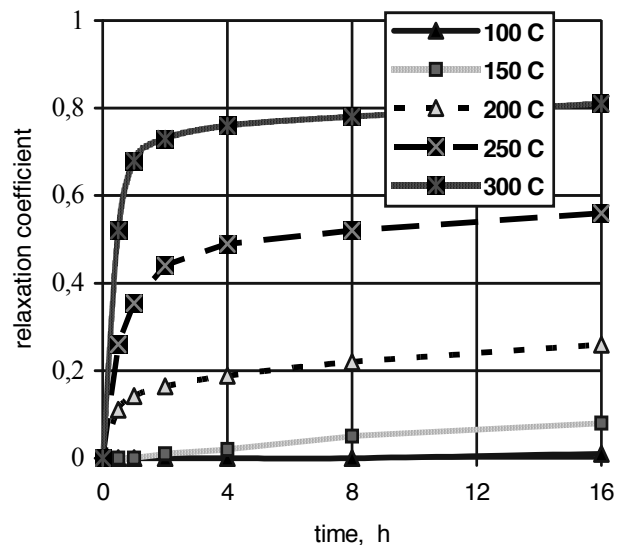


Fig. 6. Influence of temperature and time of annealing on stress relaxation coefficient of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy samples

The obtained results indicated that the relaxation processes, especially relating to reduction of free volumes and the associated stress relaxation processes are already existing at relatively low

temperatures of 100 and 150°C at extended annealing time from 8 to 16 hours.

It is also a reason of partial change in fracture morphology, where in the ductile fracture of samples visible veins are more fine and dense than in the material in as-cast state. There is no significant changes in strength of studied alloy.

Annealing at 200°C caused at a beginning the increase of strength of tested alloy to 1880 MPa, which studied ribbons achieved after 2 hours of annealing. This is the greatest strength of samples, which was obtained for the investigated alloy. The increasing of the annealing time caused decreasing of the strength to about 1630 MPa for 16 hours. The increase of strength should be considered as important result, because it is higher than the measurement error and standard deviation. The increase of strength is likely to be associated with significant reduction of free volume, increasing of density with maintaining the amorphous structure.

Further decrease of strength of studied material with increasing of annealing time is associated with topological and chemical changes and the close arrangement of atoms before beginning of the crystallization process. Plasticity of the alloy is still high and it enables bending the sample to contact their surfaces without cracking ($\varepsilon = 1$). The high plasticity of alloy was confirmed by observing the dense shear bands visible on the outer surface of the tested ribbon close to fracture morphology of strength samples – Figure 7.



Fig. 7. Shearing bands located on outer surface of sample near fracture of strength samples after annealing at 200°C/8h, (SEM, magn. 5 000x)

Investigations of fracture morphology of samples showed the occurrence of mixed fracture, which is characteristic for ductile and “river” areas and a smaller share of areas with the scaly fracture (Fig. 8). This morphology indicates a relaxation process in studied material, which is resulted in the further activation of heat treatment with the loss of high plasticity of material. Structure of the material is still amorphous.

Stress relaxation tests show that in the annealing temperature of 200°C, there are significant processes causing the reduction of internal stress from the time of annealing of less than 2 hours (after 2 hours of annealing $\eta = 0.16$). These processes also confirmed the structural relaxation in studied material, which caused the changes in the atomic configuration in the areas of short-range arrangements (topological nature of amorphous structure). After 16 hours of annealing the relaxation rate reaches a value of $\eta = 0.26$ (Fig. 6.).

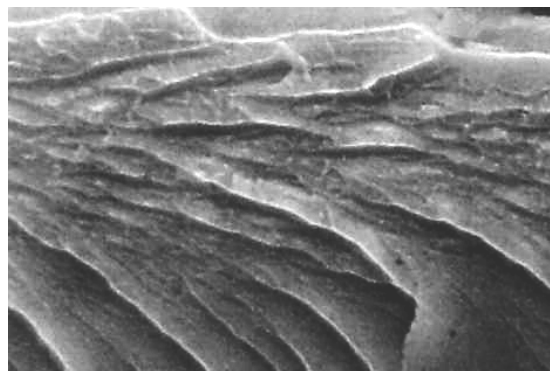


Fig. 8. Fracture morphology of strength sample after annealing of 200°C/16h of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy (mixed scaly fracture with locally “river” patterns, SEM, magn. 5 000x)

The nature of the observed changes in stress relaxation as a function of temperature and time indicates that obtained dependence has the logarithmic behavior in function of time. The process of relaxation is accompanied by changes in the geometry of samples (Fig. 5). Mainly due to reduction of free volume and the ordering of topological and chemical structure.

The increase of annealing temperature to 250°C caused very significant changes in the tested mechanical properties. The increasing of annealing time significantly reduces the strength and plasticity of studied material (Figs. 9, 10). After annealing for 16 hours - the tensile strength decreases to the value of 1360 MPa, while ductility is reduced to the value of $\varepsilon = 0.69$ after 2 hours of annealing and $\varepsilon = 0.18$ after 16 hours, consequently.

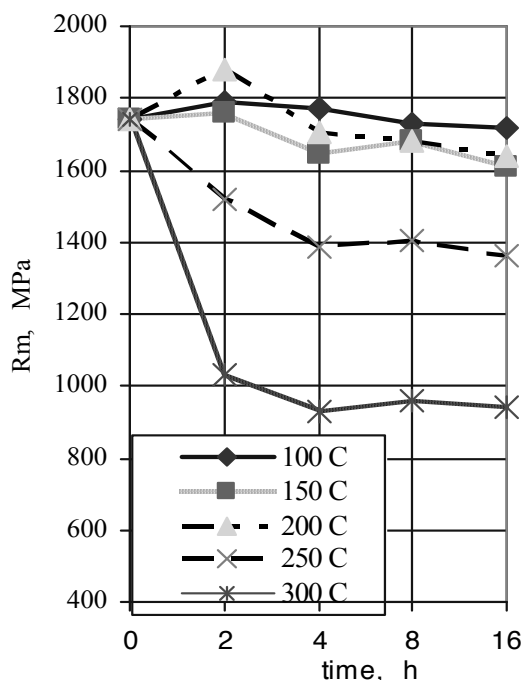


Fig. 9. Influence of annealing time on tensile strength changes of tested ribbons at temperature range from 100 to 300°C

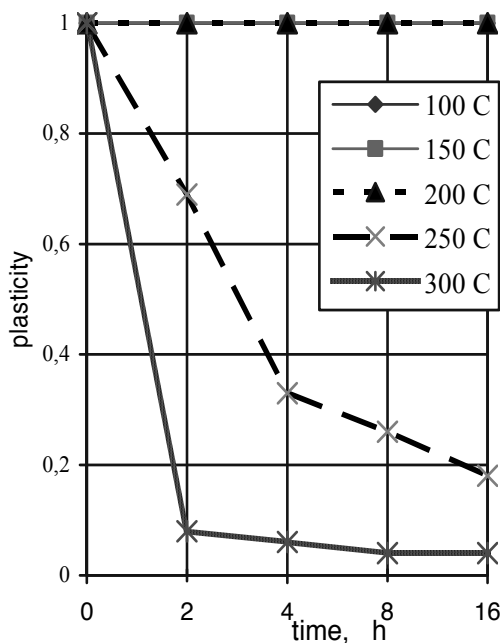


Fig. 10. Influence of annealing time on plasticity changes of tested ribbons at temperature range from 100 to 300°C

The study of fracture morphology revealed the occurrence of fine “river” patterns of strength samples (Fig. 11) and scaly fractures after increasing the time of annealing, which are typical for metallic glasses with reduced ductility. Additionally, on a part of the surface of smooth scales, it was observed a dense matrix of very fine “veins” at high magnification, which is a residue of the plastic deformation of samples (Fig. 12).

A continuation of the structure study of tested alloy revealed an amorphous structure, although the TEM researches showed a more intense contrast, which may indicate the beginning of the crystallization process (especially after annealing for 16 hours). However, the crystalline nucleation in this state was not found. The observed changes in mechanical properties and fracture morphology of the alloy are associated with relaxation phenomena of amorphous structure.

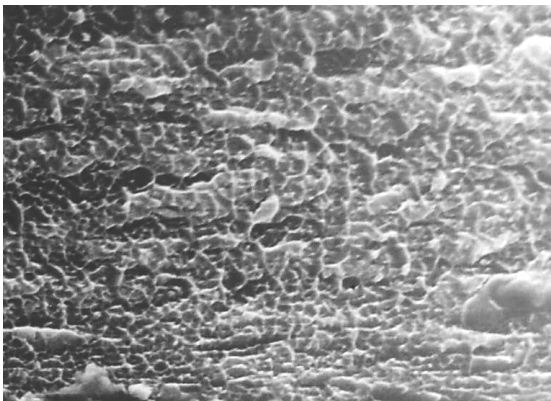


Fig. 11. Fracture morphology of fine “river” patterns of strength sample after annealing at 250°C/2h, (SEM, magn. 5 000x)

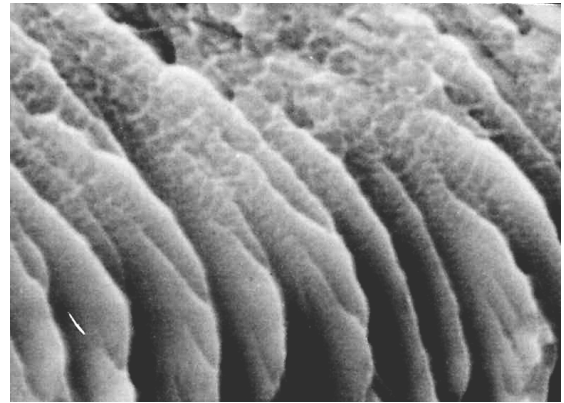


Fig. 12. Scaly fracture morphology of strength sample after annealing at 250°C/16h with locally microveins matrix, (SEM, magn. 10 000x)

The study of stress relaxation and dimensional changes of samples showed that after 16 hours of annealing process it was observed more than 50% reduction of internal stresses as a consequence of relaxation processes. Moreover, dimensional changes were also observed in tested samples, although after the annealing of 8 hours, they are no more significant. Changes in tensile strength and yield strength for the samples after annealing at 250°C and time up to 16 hours could be associated with the occurrence of structural relaxation phenomena and some structural effects before the crystallization.

After annealing at 300°C it was observed a high reduction of strength (R_m) from value of 1030 to 940 MPa and a plasticity ($\varepsilon = 0.04$) – Figures 9, 10. This decrease is related to the partial crystallization of the amorphous matrix.

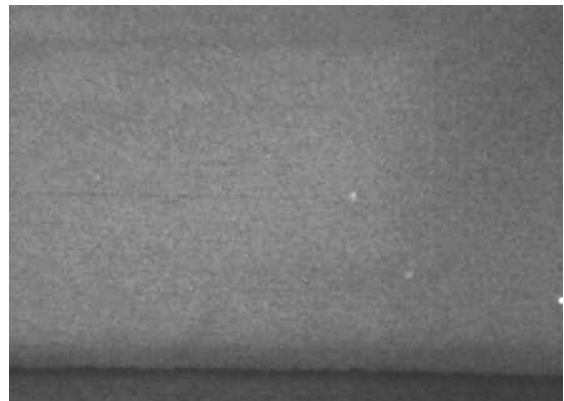


Fig. 13. Smooth and brittle fracture morphology of strength sample with very fine roughness of surface after annealing at 300°C/2h, (SEM, magn. 5 000x)

Surface of studied fracture is smooth and characteristic for a brittle material with very fine roughness, which is likely derived from the borders of crystallites – Fig. 13. The annealing temperature of 300°C caused the initiation of the crystallization

process by formation α -Fe crystallites in an amorphous matrix. The diffraction pattern presented reflections from a single crystalline of α -Fe phase (Fig. 14).

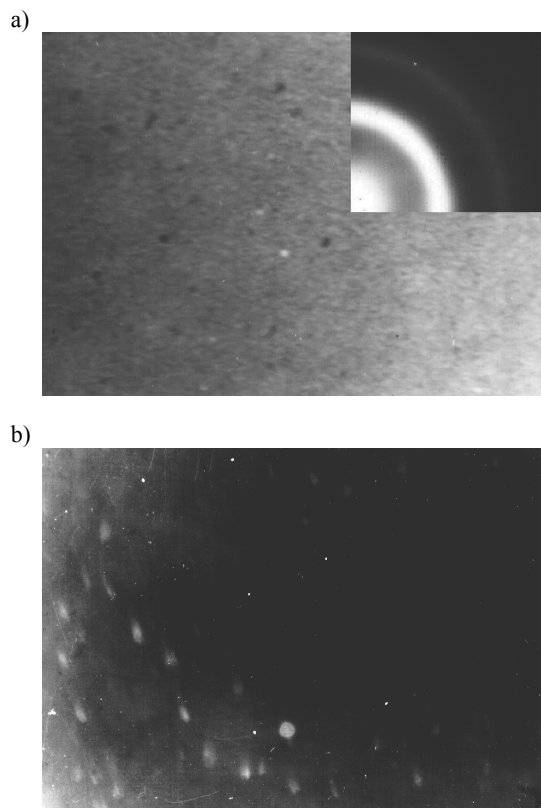


Fig. 14. a) Transmission electron micrograph and electron diffraction pattern of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy after annealing at $300^\circ\text{C}/4\text{ h}$ (very fine spherical crystalline of α -Fe in amorphous matrix, TEM, magn. 50 000x); b) Dark field from reflection of (112) α -Fe, TEM, magn. 150 000 x

This phase occurs in a small share of the main crystallization product of $\text{Fe}_{78}\text{B}_{13}\text{Si}_9$ alloy after annealing at 300°C . The remaining volume of structure has the amorphous phase. After the increase of annealing temperature it could be observed a formation of very fine Fe_2B borides near spherical crystallites of α -Fe phase. At the same moment during the annealing process it could be also determined a significant reduction of internal stress, which after 2 hours has a value of $\eta = 0.73$ and after 16 hours reaches a value of $\eta = 0.81$, consequently. The annealing temperature of 300°C led to the initiation of crystallization, which is also accompanied by further dimensional changes of the samples (Fig. 5).

4. Conclusions

The studied ribbons of $\text{Fe}_{78}\text{B}_{13}\text{Si}_9$ alloy in as-cast state exhibited an amorphous structure. In this state, samples have a tensile strength with a value of 1740 MPa and a high plasticity

($\varepsilon = 1$). The fracture morphology achieved in a tensile test is mixed with “river” patterns.

The beginning of the structural relaxation was revealed after annealing at 100 and 150°C , especially after long annealing times of 8 and 16 hours. It was observed that fractures of samples have a more diverse morphology than ribbons in as-cast state. The structural relaxation process was confirmed by examination of dimensional changes of samples.

The increase of annealing temperature to 200°C caused initial increasing of strength, which is probably associated with a significant loss of free volume and the increase of density with maintaining of amorphous structure. The decrease of strength is probably associated with further topological and chemical changes of short-range arrangement of atoms. The changes in fracture morphology were observed. The process of structural relaxation is accompanied by changes in the geometry of samples.

The increase of annealing temperature to 250°C caused very significant changes of the strength and ductility of samples. The fracture morphology observations revealed the existing of areas with fine veins and scaly fractures. The study of stress relaxation and dimensional changes of tested samples showed more than 50% reduction of internal stresses after 16 hours of annealing. The observed changes of properties could be associated with relaxation processes of metallic glass.

Significant changes in the structure and properties of the alloy was observed after annealing at 300°C . The reduction of strength and the increase of fragility of samples was determined. This decrease is associated with extending of the structural relaxation and beginning of crystallization process by formation of small crystallites of α -Fe phase in amorphous matrix. This process is also accompanied by dimensional changes of samples and a significant stress relaxation processes.

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