



Influence of thermal activation on changes of mechanical properties and tensile fracture morphology surface of Ni-base metallic glasses

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ABSTRACT

Purpose: The paper presents a structural relaxation and crystallization process of $\text{Ni}_{68.7}\text{Cr}_{6.6}\text{Fe}_{2.65}\text{Si}_{7.8}\text{B}_{14}\text{C}_{0.25}$ metallic glasses. The $\text{Ni}_{68.7}\text{Cr}_{6.6}\text{Fe}_{2.65}\text{Si}_{7.8}\text{B}_{14}\text{C}_{0.25}$ metallic glasses were produced by the CBMS method for two different conditions of casting at different cooling rate.

Design/methodology/approach: The relaxation and crystallization of $\text{Ni}_{68.7}\text{Cr}_{6.6}\text{Fe}_{2.65}\text{Si}_{7.8}\text{B}_{14}\text{C}_{0.25}$ metallic glasses by methods: mechanical tests, scanning electron microscopy (SEM) and internal friction (IF) were studied.

Findings: The investigation showed that conditions of vitrification (different, but higher than critical cooling rate) influence on different course of elementary structural relaxation processes during thermal activation.

Research limitations/implications: The differences in temperature of the beginning of elementary relaxation processes and crystallization processes of alloy, in relation to strip thickness were disclosed.

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

Originality/value: The paper presents those conditions of vitrification influence on different course structural relaxation processes during thermal activation.

Keywords: Amorphous materials; Relaxation; Crystallization of metallic glasses; Mechanical properties; Fracture morphology

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MATERIALS

1. Introduction

The metallic glasses, obtained in a result of fast cooling from the liquid state, show thermodynamic unbalance. Thermal activation of metallic glasses leads to the structural changes, which in the final stage is the crystalline state. Conditions of metallic glasses production can influence on the course of structural relaxation [1-6] and crystallization [7-11, 14-19] during thermal activation.

The conditions of metallic glasses production have essential influence on their amorphous structure - in different degree of ordering or disordering of amorphous structure. Therefore, the condition of liquid alloy solidification decides about phenomena which proceed in thermal activation of metastable metallic glasses and its structure and properties. It is possible to get different states of metallic glasses induced by fast cooling of a liquid alloy, at different speeds of cooling.

In the present work, the course of structural relaxation process of the metallic glass like $\text{Ni}_{68.7}\text{Cr}_{6.6}\text{Fe}_{2.65}\text{Si}_{7.8}\text{B}_{14}\text{C}_{0.25}$ for two strips of the same chemical composition, but of different transverse sections - and thus, probably of a different vitrification state, was introduced. By a method of bend and tensile tests and scanning electron microscopy (SEM) as well as a method of the internal friction (IT), the structural relaxation process was studied. In result of investigations of metallic glasses after thermal activation, structural relaxation process runs differently in dependence on initial amorphous structure state (the state of vitrification) produced as a result of application of different cooling rates for a liquid alloy providing its vitrification conditions.

2. Experimental procedure

The material for investigations was $\text{Ni}_{68.7}\text{Cr}_{6.6}\text{Fe}_{2.65}\text{Si}_{7.8}\text{B}_{14}\text{C}_{0.25}$ alloys appointed according to American Welding Society as BNi2 Material, cast as metallic glass in a form of strips with dimensions:

- thickness 0.030 mm, width 5 mm - appointed as a_T
 - thickness 0.045 mm, width 3 mm - appointed as a_F ,
- on surface of turning chromic copper drum. Casting of the strips was conducted in The Institute of Engineering and Biomedical Materials of The Silesian University of Technology and it was conducted at the pressure of gas stuffing 70 kPa and at two circumferential cooling rates of the drum 20 and 24 m/s. The strips were produced by a „chill - block - melt- spinning” method- this is a method of continuous casting of the liquid alloy.

In the works [12, 13, 14] the exact data concerning production of the studied alloy were presented. Manufactured metallic glasses in the form of strips, appointed as a_T and a_F showed large plasticity as-cast ($\varepsilon = 1$; and amorphous structure) which was confirmed by x-ray and electron diffraction.

The tensile test, at room temperature, was conducted using samples in "as quenched" state and as well as after pre-heating to the following temperatures 373, 423, 473, 523, 573, 623, 673 K and warming up by 2 h, and afterwards final cooling down. The thermal processing, to exclude oxygenation of the samples, was conducted in vacuum heaters. The comparative material was a material in "as quenched" state.

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.

That is the number of tests which was carried out by 20 samples for each state. The samples of strips sections about 120 mm were fixed in flat jaws facing rubber. Distance among grips of tensile machine was 50 mm, and the speed of machine beam was 5 mm / minute.

To eliminate the influence of the micro-inequality of strips banks (geometry of samples) on measurements results, in the statistical analyse, 5 running away from the measurement results for every series of samples were not taken into account. The investigations results have been worked out with the help of the STATGRAPHICS version 5.0 software.

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 jaws up to durable rise of maximum deformation or to a crack creation. The plasticity deformation answering the yield stress was calculated from the expression:

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

where:

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

t - strip thickness

The strips sections about 120 mm long for the studied metallic glass were subjected to thermal treatment for selected conditions of heating in the range of temperatures to 673 K.

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 1000 to 3500 times.

The investigation of internal friction was conducted at room temperature. The samples were pre-heated to higher and higher temperatures, and afterwards, cooled down to the room temperature, in which the vibrations frequency measurement was carried out. In the temperatures range to 800 K at constant pre-heating rate was 2 K / min., the function of frequency f_{300K}^2 in dependence on pre-heating temperature was marked. Investigation was conducted in vacuum using an inverse torsion pendulum of Kê type. In the relative dependence of elasticity module changes, G is proportional to changes of the trembling frequency of sample $G \sim f^2$.

3. Results

The results of strength investigations R_m with standard deviations of the strips a_T and a_F of the studied alloy $\text{Ni}_{68.7}\text{Cr}_{6.6}\text{Fe}_{2.65}\text{Si}_{7.8}\text{B}_{14}\text{C}_{0.25}$ as the function of heating temperature are presented in Table 1. The curves of the strength dependence R_m versus heating temperature for both strips show the different

course what was illustrated in the Figs. 1 and 2. In a state directly after casting (aq) the sample a_T possesses strength equalled to 1634 MPa. The strength of the sample a_T in a range of warming temperatures to 523 K shows the growth to the state as quenched (aq) and achieves maximally 1837 MPa. After warming to higher temperatures, the strength of the strips diminishes, after being pre-heated to 673 K of the similar level strength as in the state of aq (Table 1, Fig.1).

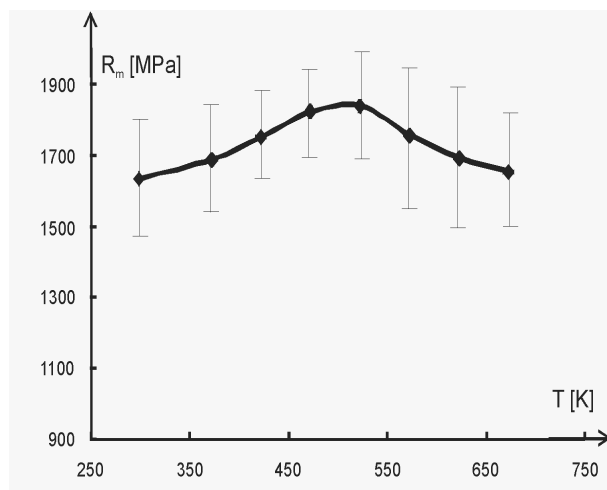


Fig. 1. The R_m dependence in function of the pre-heating temperature for the a_T sample

The dependence of changes of the strength R_m versus warming temperature for strips - a_F (the Table 1, Fig. 2) is different. As being in the aq state, the strength of a_F strips is higher and it carries out 1722 MPa. In the range of warming temperatures to 473 K, very low, unimportant increase of the strength is observed, which is containing in range of standard deviation. For higher warming temperatures the strength of strips gets lower and the course of the R_m curve changes is similar to the appointed one for strips a_T (Table 2, Fig. 2.)

The standard deviations calculated for a_T strips as well as a_F showed comparatively high values, what is connected with interaction of the micro-unevenness of the strips banks as well as the large sensitivity of measurements for alignment during the tensile test.

Table 1.

The results of mechanical properties determined at different temperatures for a_T samples

T [K]	300	373	423	473	523	573	623	673
R_m [MPa]	1634	1687	1750	1821	1837	1756	1691	1652
Standard dev. σ [MPa]	163	148	132	139	174	211	192	169

Table 2.

The results of mechanical properties determined at different temperatures for a_F samples

T [K]	300	373	423	473	523	573	623	673
R_m [MPa]	1722	1754	1766	1757	1654	1584	1532	1509
Standard dev. σ [MPa]	146	130	128	134	156	179	144	158

In spite of this, it was noticed a clear difference in changes of R_m in warming temperature function for studied material about different degree initially (after casting) relaxing of amorphous structure.

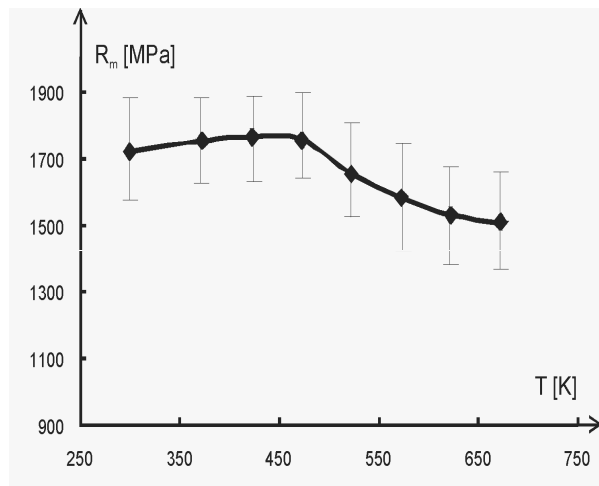


Fig. 2. The R_m dependence in function of the pre-heating temperature for the a_T sample

The investigation of strips fracture, in as-quenched state, following the tensile tests showed that the fractures have the characteristic river morphology with the well developed river structure. The fracture structure of both a_T and a_F ribbons are similar enough, however, in the exact analysis small differences can be noticed in morphological respect. In as-cast samples for a_T state (Fig. 3) observed on the fractures, threads are fatter, more prominent and thinner, in comparison to a_F samples (Fig. 4) where the threads are thicker and smaller.

The warming of the samples a_T at temperature 373 K did not show any influence on morphology of fracture in comparison to morphology of fracture for sample in as - quenched state.

Structure of this fractures (Fig. 5) shows similarly fat, expressive and thinner threads as for as - quenched state.

These differences of density and river morphology of fracture for a_T and a_F samples exist up to pre-heating temperature 473 K (Figs. 6 and 7).

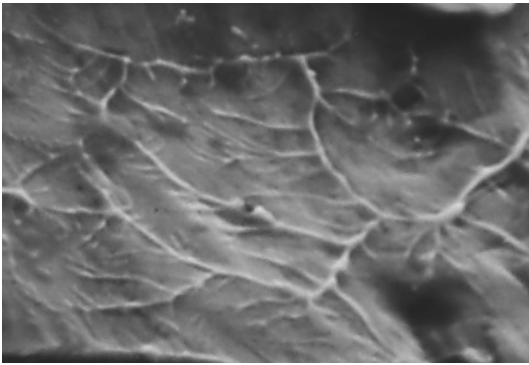


Fig. 3. Tensile fracture surface of the a_T samples in as-quenched, 2500x

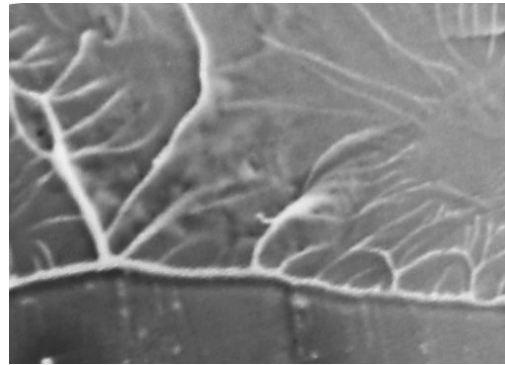


Fig. 6. Tensile fracture surface of the a_T samples annealed at 473 K, 3000x

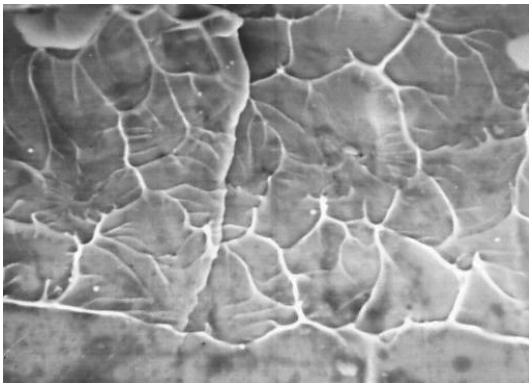


Fig. 4. Tensile fracture surface of the a_F samples in as-quenched, 2500x

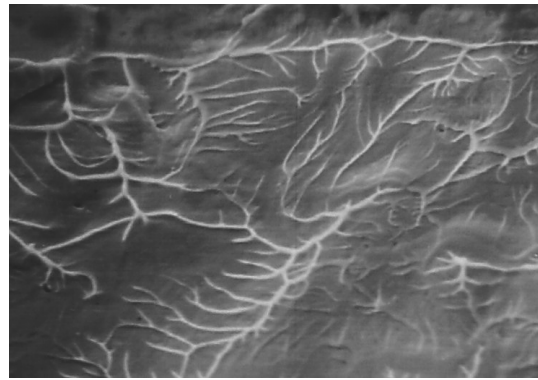


Fig. 7. Tensile fracture surface of the a_F samples annealed at 473 K, 2800x

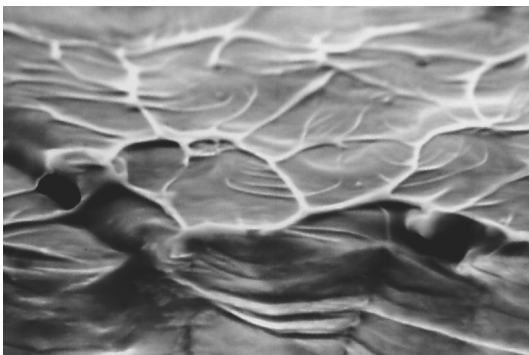


Fig. 5 Tensile fracture surface of the a_T samples annealed at 373 K, 2200x

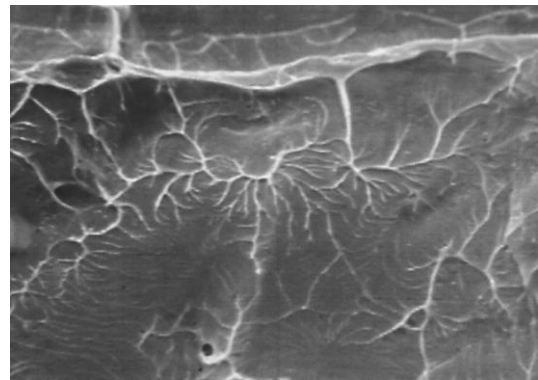


Fig. 8. Tensile fracture surface of the a_T samples annealed at 573 K, 2800x

Only warming of samples at 573 K and higher, in a result of very advanced and already the same relaxing amorphous structure both a_T and a_F strips causes, that fractures are very similar and characterize the thread structure (river fractures) with numerous, small and complex threads (Fig. 8).

After warming of the both a_T and a_F strips at the temperature of 623 K, on predominant areas of strips fractures of very numerous smooth scales (Fig. 9), and moreover, in few areas of remainder of changes threads were observed After strips

pre-heating at 673 K the thread areas on their fractures disappear entirely. The strips become very brittle and the fractures are characterized by the presence of uneven scales as well as the areas of homogeneous changes - the unevenness of surface (Fig. 10).

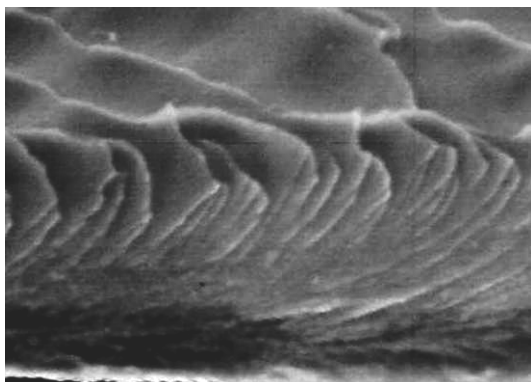


Fig. 9. Tensile fracture surface of the a_F samples annealed at 623 K, 3200x

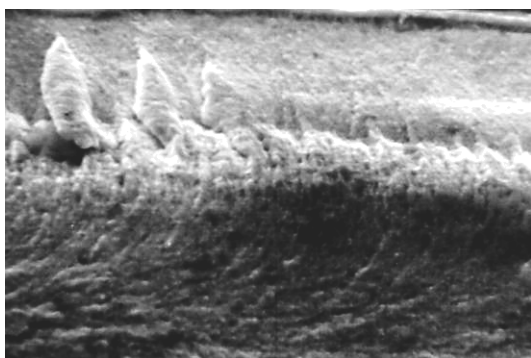


Fig. 10. Tensile fracture surface of the a_T samples annealed at 673 K, 2000x

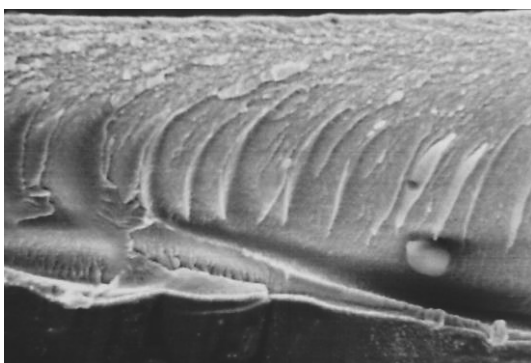


Fig. 11. Tensile fracture surface of the a_F samples annealed at 773 K, 1500x

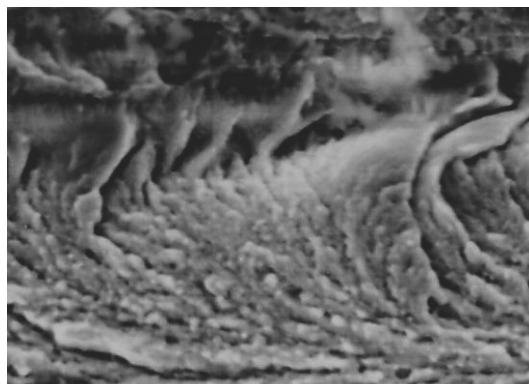


Fig. 12. Tensile fracture surface of the a_T samples annealed at 873 K, 3200x

The strips warming at 773 K causes that the areas of fractures containing scales are more developed and smooth, and the part in it possesses clear regular unevenness of the material surface being in already partially crystallized state (Fig. 11). The samples after pre-heating at 873 K possess scale morphology, however with enough developed surface (Fig. 12), it can be connected with further more partial crystallization of alloy.

The plasticity investigations of the strips showed that both a_T and a_F as-quenched strips are characterized by high plasticity directly characteristic for amorphous structure ($\epsilon = 1$). The strip warming in the range of temperatures to 573 K does not lead to loss of high material plasticity however, a violent loss of plasticity for higher temperature follows for both a_T and a_F strips. The plasticity changes of the strips a_T and a_F in dependence on warming temperature was presented in the Figs. 13, 14. The warming of the strips at the temperature 623 K, and particularly above 673 K, causes considerable brittleness of the material ($\epsilon = 0.02$).

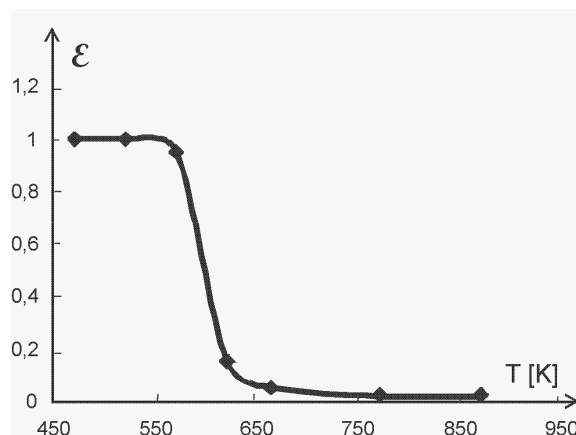


Fig. 13. The dependence of plasticity in function of the temperature of the a_T sample pre-heating

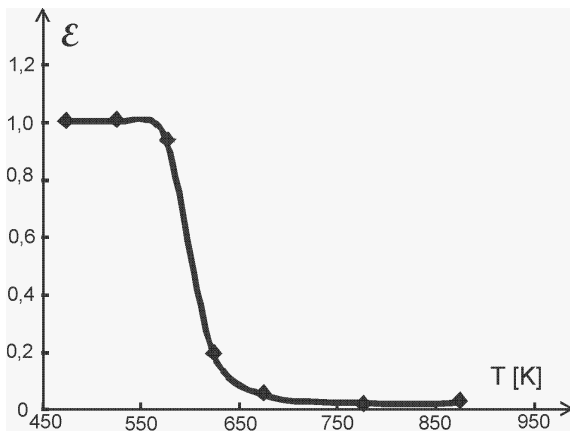


Fig. 14 The dependence of plasticity in function of the temperature of the a_T sample pre-heating

The course of elasticity G module changes (changes of f^2 function) appointed by the Kê pendulum type, at room temperature for both a_T and a_F strips, in a function of maximum temperature to which the sample was warmed up, is presented in Figs. 15 and 16. The conducted investigations showed the course of elasticity modulus changes considerable differences in dependence on warming temperature of a_T and a_F strips. The temperature dependence of elasticity module appointed for a_T strip shows, after 423 K is exceeded, a clear fall of module value achieving the minimum temperature 550 K (Fig. 15). After samples are pre-heated at higher temperatures, considerable growth of elasticity module value follows, observed for a_T strip, until warming up to the temperature of 850 K. Apart from this temperature, further changes of elasticity module were observed which are established on a constant level.

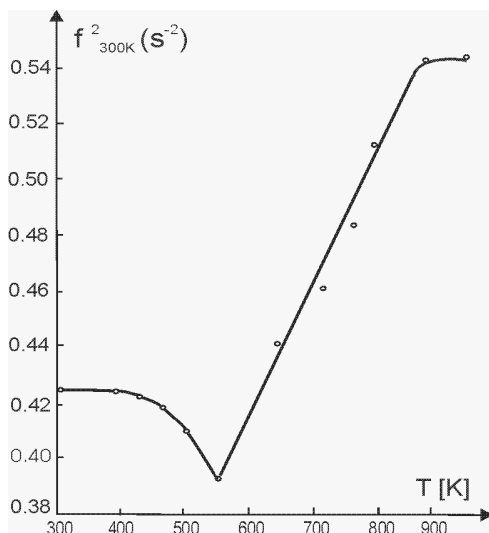


Fig. 15. Relation of elasticity module f^2_{300K} for temperature 300K in function of the maximum heating temperature of the sample a_T

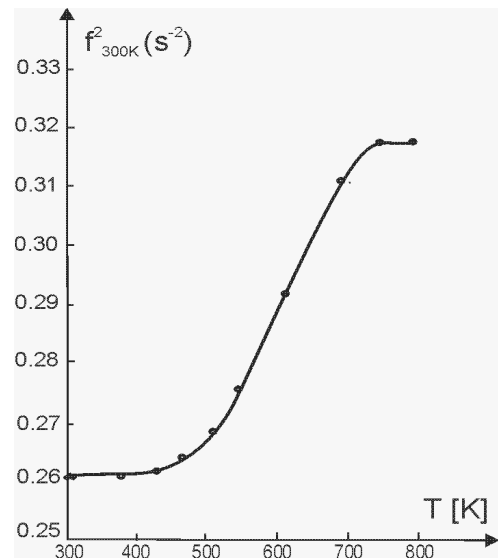


Fig. 16. The dependence of the elasticity module f^2_{300K} in the temperature of 300K in function of the maximum temperature of a_F sample pre-heating

The curve of elasticity module changes at room temperature appointed as a change of function f^2_{300K} for the a_F strip warmed up to different temperatures is presented in the Fig. 16. In the warming temperatures range to about 423 K, a change of elasticity module value almost does not step out. Apart from this warming temperature, initially mild, and later in the range of 523 to 723 K linear, steep growth of elasticity modulus was observed. The further rise of temperature to 800 K does not cause the growth of elasticity module value any longer for a_F samples (Fig. 16).

4. Discussion about results

The conducted investigations, using varied, supplementary investigative methods, as well as received results allowed to determine the thermal activation influence on mechanical properties changes and the studied morphology of fractures for the metallic glasses a_T and the a_F strips. The a_T and a_F strips with initial amorphous structure, yet differed by casting conditions and thickness which influenced on initial stages of structural relaxation, what was the symptom of the observed changes particularly in fracture morphology as well as the strength change versus warming temperatures of the samples. The presented work is the continuation of the earlier investigations in which the processes of the structural relaxation and the crystallization of $Ni_{68.7}Cr_{6.6}Fe_{2.65}Si_{7.8}B_{14}C_{0.25}$ metallic glass were analyzed for the a_T and the a_F samples as well as the characteristic temperature ranges of the setting structural changes [5, 14 -17].

Produced by the casting method on rotating copper drum the $Ni_{68.7}Cr_{6.6}Fe_{2.65}Si_{7.8}B_{14}C_{0.25}$ a_T and the a_F metallic glasses strips showed high plasticity as well as amorphous structure in the as

quenched (Figs. 13 and 14). The fractures investigations of samples after the tensile test showed characteristic river fractures for ductile metallic glasses, however differing thickness of threads in a moderate degree. Smaller arrangement of threads stepped out for the a_F strip what can be also shown on different (larger) stickiness of metallic glass after casting according to initial stage of the structure relaxing (Figs. 3 and 4). In as-quenched state, the strength of strips for a_T and a_F is performed correctly - 1634 MPa as well as 1722 MPa, what except influence of thickness, show also influence of initial state of structure.

Together with growth of warming temperature, the irreversible changes in structure of the studied metallic glasses follow. These changes were observed particularly in the internal friction investigations determining the relative changes of elasticity module. First changes of physical properties of the studied samples have already been set after warming to the temperature about 423 K. The measurement of elasticity module $f_{300K}^2(T)$ appointed to a_T (Fig. 15) samples showed that in the growth of temperature, the elasticity module rises lower values, stepping out at minimum 550 K, the temperature raising further leads to a growth of module. These changes testify about setting intensive processes of relaxation of amorphous structure. The curve of elasticity module is characterized by different course $f_{300K}^2(T)$ appointed to a_F strip (Fig. 16). After warming the samples above the temperature 423 K, elasticity module raises higher and higher values, which shows that after casting, the material with amorphous structure was able to be strongly relaxed, and the crystallization of material changes of module growth may be connected with previous processes directly.

Stepping out differently for a_T and a_F strips, relaxing processes also influenced on a change of strength of strips. The a_T strip in the range of warming temperatures to 523 K shows the R_m growth about 200 MPa in relation to aq state what can be connected with a running relaxing process and atrophy of free volume. After warming up to higher temperatures, strength of strips diminishes. The dependence of strength R_m changes versus temperature for a_F strip is different. In the aq state the strength of a_F strip has already been higher and it has been reached 1722 MPa and so low changing level keeps the temperature of 473 K which shows that very low relaxing resulting from considerable advanced processes changes of structure, already set in this range of temperatures directly after casting. For higher warming temperatures the strength of strips gets lower and the course of the R_m curve changes is similar to appointed ones to a_T strips, however the R_m level is lower, which should be translated on measurements result with influence on strip thickness. It should also infer that the differences in course of relaxing processes among a_T and a_F strips concern the range of temperatures to 523 mainly for a_T as well as 473 K for a_F .

Above these temperatures for both types of strip, the relaxing processes run similarly leading to higher temperatures qualitatively (above 650 K) to the beginning of crystallization process. They also confirm the change of strips plasticity to similar course for which it was observed. They, in the range of temperatures to 573 K of the strip, are characterized by equally high characteristic plasticity for amorphous structure ($\epsilon = 1$), and then, in higher temperatures, particularly above 673 K, violent fall of plasticity, what already concerns the stages directly before crystallization and the start of crystallization.

Structure of both strips fractures - a_T and a_F are enough similar, however, the exact analysis has shown small differences - in

samples after casting for a_T state on fracture where more prominent and thinner fatter threads were observed in comparison to a_F samples which show smaller relaxing structure. Such differences in thickness and the appearance of threads in morphology of fractures for a_T and a_F samples keep the warming temperatures at 473 K. Only warmed samples at 573 K result on very advanced and already equal relaxing amorphous structure, it causes that both a_T and a_F strips get similar fractures which are marked approximately in appearance to the river structure about enough numerous, small and complex threads. Strips in this state are still plastic ($\epsilon = 1$). From this warming temperature fractures for observed types of samples are among them undistinguishable. The essential change of fractures morphology has been observed after pre-heating both a_T and a_F strips in the temperature of 623 K and at the beginning of high plasticity loss of strips following simultaneously. Very numerous, smooth scales on predominant areas of fractures were observed, and in a few areas of remainder of threads changes. Warming the strips at 773 K causes that the areas of fractures with scales are more developed and smoother, and the part of it possesses clear regular unevenness of surface of material being in an already partially crystallized state. Samples after pre-heating in 873 K possess scales morphology, however with enough developed surface, what can be connected with more partial crystallization of alloy.

The presented investigations results testify that a setting change of property in the studied temperature range and morphology of samples fractures is connected with structural relaxation and the crystallization processes of strips. These processes occur in low-temperature range differently (to 473 K) for a_T and a_F strips which may be connected mainly with changes of free volume, which probably in amorphous alloy undergoes the changes and the atrophy in the process of structural relaxation. The growth of alloy thickness on a stage of relaxation in result of atrophy of free volume can particularly be cause of growth of elasticity module, growth of alloy strength as well as growth of stickiness which manifests in received results of investigations.

Therefore, the observed changes in studied alloys at this temperature range particularly to 473 K to bind topological structural relaxation, depending on change relative positions of atoms in amorphous structure cause displacement and atrophy of free volume.

Based on findings' results, it is possible to present a different course of structural relaxation for both a_T and a_F samples produced in different conditions.

For a_T samples which were produced as thinner strips and at higher cooling rates during casting, one can distinguish two stages of structural relaxation, divided by clear temperature border line at the temperature of 550 K:

Stage 1 - the range of temperatures close to room temperature up to 550 K (523 K), after clear lowering the elasticity module of $f_{300K}^2(T)$ the minimum steps out. The TRSO in this stage steps out - the topological structural relaxation is connected with intensive displacement and atrophy of free volume; (significant change of elasticity module as well as R_m strength growth).

Stage 2 - close to the range of temperatures above 550 K for the chemical structural relaxation about 650 K. In this stage chemical structural relaxation (CSRO) follows, depending on relations of a close range change, the setting among different chemical components and the order of atoms given by a type of component.

For a_F strip produced at lower cooling rate, a_T state answers the amorphous structure about considerably advanced relaxing processes. Far more structural relaxation runs in temperature range particularly above 473 K. The lack of clear, characteristic points on curves of temperature dependence of elasticity module E_{300K}^2 (T) in range of lower temperatures as well as nearly lack of changes of R_m strength in function of warming temperature testifies that topological structural relaxation (TRSO), connected with displacement and atrophy of free volume is not occurring or occurring in considerably lower degree in comparison to changes in a_T sample because the conditions of cooling while producing a_F strip have made such incident relaxing processes already possible.

Based on analysis of the presented results it is possible to conclude that a_F sample had more relaxed exit structure than a_T sample. Comparing the thickness of studied strips, - the a_F samples thickness is higher (0.045 mm) than the a_T samples (0.03 mm), as well as the parameters of production - the lower cooling rate for the a_F sample in comparison to a_T , we can confirm regularity of a statement about different initial stage of relaxing structure in process of the metallic glass production or different amorphous state of structure. Those investigations result were confirmed by the investigations results in this paper [5, 15 -17]

The conducted investigations showed that the production conditions of metallic glasses influence the geometry of the received metallic strips and moreover, differentiating the cooling rate of metallic liquid, they are the cause of changes in course of crystallization process during thermal activation of the received metallic glasses.

The obtained results allow to affirm that the course of relaxation and crystallization process, both for a_T and a_F strips, the glass metallic $Ni_{68.7}Cr_{6.6}Fe_{2.65}Si_{7.8}B_{14}C_{0.25}$ in result of thermal activation runs differently. In relation to amorphous state of metallic glass structure received in result of cooling from the state of liquid alloy at different cooling rates (the different parameters of production) the different strength of strips was received as well as the morphology of fracture. The affirmed changes are the result of diverse structural relaxation processes of metallic glass.

The above mentioned investigations show that the conditions of production of metallic glass have the influence on course of structural relaxation, and also on course of initial stages of relaxation and crystallization of alloy. This conclusion was confirmed by investigations conducted in these works [5, 14-17].

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