Vol. **113** (2008) *ACTA PHYSICA POLONICA A* No. 1

Proceedings of the CSMAG'07 Conference, Košice, July 9–12, 2007

Structural Relaxation of Ni–Si–B Amorphous Ribbon

A. Juríková, K. Csach, J. Miškuf

Institute of Experimental Physics, Slovak Academy of Sciences
Watsonova 47, 040 01 Košice, Slovakia

and V. Ocelík

Department of Applied Physics, Materials Science Centre and Netherlands
Institute of Metals Research, University of Groningen
Nijenborgh 4, 9747 AG Groningen, The Netherlands

The structural relaxation of the Ni–Si–B amorphous ribbon was studied by means of differential scanning calorimetry and thermomechanical analysis. It was shown that considerable length changes associated with reversible structural relaxation were revealed after a previous creep applied at higher temperatures.

PACS numbers: 61.43.Dq, 62.20.Hg

1. Introduction

Some amorphous alloys represent a class of metallic materials with unique mechanical and soft magnetic properties. They are unstable from a thermodynamic point of view and annealing them at elevated temperatures leads to structural relaxation manifesting itself in changes of various physical properties [1–3]. Atomic disorder and defects of different levels play the important role in magnetic properties of metallic glasses [4–6].

Glassy alloys with a high Ni content were found to exhibit a glass transition before crystallization. The combination of high glass-forming ability and good mechanical and soft magnetic properties of the Ni-based glassy alloys indicates their perspective applications [7]. The homogeneous deformation ability is influenced by the structure of amorphous alloys, namely by the amount and mobility of defects. Therefore creep and creep recovery experiments can help understanding these phenomena [8, 9]. The aim of the presented work was to find out how the creep applied at different temperatures influences the reversible structural relaxation of the Ni-rich amorphous alloy.

2. Experimental details

An amorphous ribbon $Ni_{77.5}Si_{7.5}B_{15}$ (at.%) with the thickness of 18.8 μm produced at the Institute of Physics in Bratislava by the melt-spinning method was used in experiments. The crystallization temperature of the amorphous ribbon, estimated by differential scanning calorimetry (DSC) for the as-quenched sample as the temperature of the first crystallization peak onset, is $T_x = 764$ K.

First, a piece of the amorphous ribbon was undergone six subsequent heating runs using a Perkin Elmer DSC 7 differential scanning calorimeter for studying the structural relaxation. The enthalpy changes in the each run were measured with the heating rate of 20 K/min up to the temperature 693 K well below the crystallization temperature T_x.

Next, a sample with the initial length 15 mm was undergone several thermal treatments using a Setaram TMA 92 thermomechanical analyser in the tension arrangement with the absolute resolution of 10 nm under a flowing nitrogen atmosphere. The initial structure was stabilized by two subsequent linear heatings up to the temperature 673 K below T_x. Such a thermal treated sample was considered as a reference one. Before next four linear heatings up to the temperature 673 K, the load on the sample producing the stress of about 100 MPa was applied and kept for the time of about 18 hours at temperatures of 470, 495, 620, and 645 K. Next two heating runs without a previous creep were carried out for checking the structure stabilization. Length changes were recorded during the each heating run with a rate of 10 K/min.

3. Results and discussion

DSC traces recorded for the same sample treated in six subsequent runs during linear heating up to 693 K are shown in Fig. 1. During the first run, structural relaxation is associated with the sample contraction, free volume annealing-out and energy releasing. This manifests itself on DSC trace of as-quenched sample as a wide exothermic peak from about 425 to 625 K. In each next run the free volume is still annealed out and more and more stable state is formed which is also accompanied with the sample contraction. The inset shows the DSC thermogram for as-quenched sample. The arrows marked T_g and T_x indicate the glass transition and crystallization onset temperatures, respectively.

Thermomechanical experiments were carried out to estimate the influence of creep conditions on structure relaxation processes. Measured length changes of the same sample during eight subsequent linear heatings up to 673 K are shown in Fig. 2. The thermal expansion curves were measured during linear heatings without the reposition of the sample. The shift of elongation curves is due to the creep deformation induced between some runs. Small differences in the temperature dependence of the elongation were observed among individual runs when the creep deformation introduced to the sample during the previous creep was small or absent.

The creep recovery contributions to the length changes at linear heating estimated by subtracting the elongation curve of the reference sample from these ones measured after creep annealing at different temperatures can be seen in Fig. 3. Nearly periodic alternations on dilatometric curves were caused by small non-

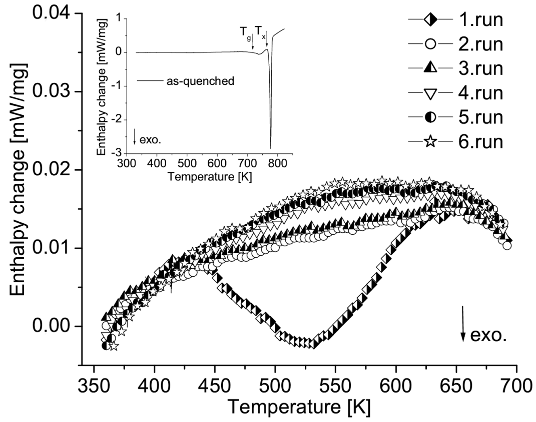

Fig. 1. DSC traces measured for the same sample in six subsequent heating runs.

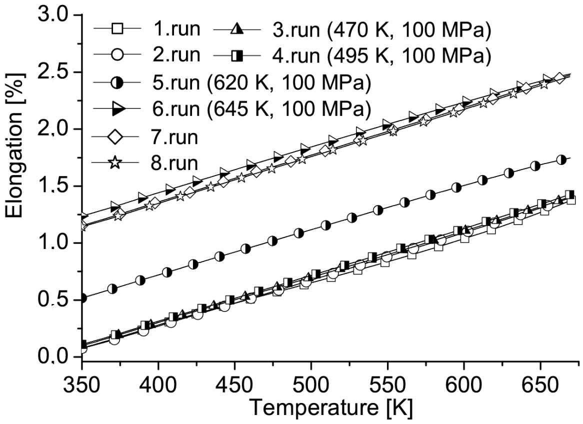

Fig. 2. Dilatometric curves measured for individual thermal treatments of the same sample. Annealing temperatures and stressess applied during creep are indicated in brackets.

linearity of heating rate due to temperature controller setting. For comparison, the temperature dependence of length changes due to structural relaxation of as-quenched sample was added. Structural relaxation during this first run is associated with considerable relative contraction of the sample starting at about 425 K that is in accordance to DSC measurements in Fig. 1.

The higher temperature of creep annealing was applied, the higher total deformation was accumulated into the sample. At creep temperatures up to 495 K the creep and creep recovery deformations were small. Increase in the creep temperature to 620 K caused increase the creep deformation to 0.38% and the creep recovery deformation to 0.1%. Further increase in the creep annealing temperature to 645 K increased in the creep-induced deformation to 0.72% as expected,

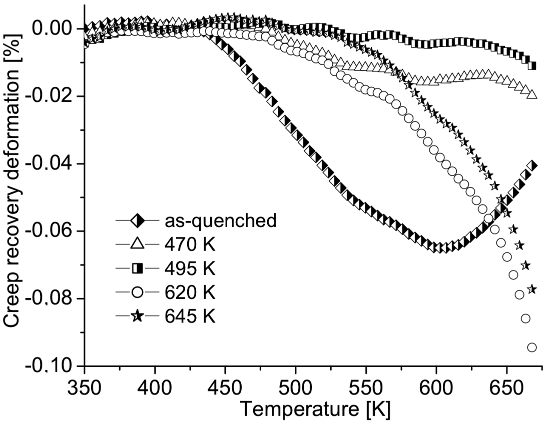

Fig. 3. The creep recovery deformation after creep at indicated temperatures and the shrinkage during structural relaxation of the as-quenched sample.

but the creep recovery deformation remained at the value similar to the value in the previous case.

In summary, the total creep deformation, consisting of the reversible and irreversible parts increases considerably with increase in the creep annealing temperature as generally expected. The temperature dependence of the reversible part (i.e. the creep recovery deformation) seems to have a saturation behaviour and so the increase in the total creep deformation with temperature is caused by the increase in irreversible contribution to the creep deformation.

Acknowledgments

The work was supported by the Slovak Grant Agency for Science – VEGA.

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