Investigation of the effect of electric pulse heat treatment of rapid-quenched Ti$_{50}$Ni$_{25}$Cu$_{25}$ alloy ribbons with a thin surface crystal layer on the structure formation from an amorphous state

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Abstract. This work is devoted to the study of the influence of electric pulse heat treatment of rapid-quenched Ti$_{50}$Ni$_{25}$Cu$_{25}$ alloy ribbons (at. %) with a surface crystal layer. The object of the study was a rapid-quenched layered amorphous-crystal ribbon with a thickness of about 30 µm and a width of about 1.5 mm, in which a thin surface crystal layer (2.5 µm) was present on the non-contact side relative to the quenching wheel. The alloy samples were subjected to electric pulse treatment by passing a single pulse of electric current through the sample with a variable duration. For comparison, samples of the alloy were obtained, crystallized by standard isothermal heat treatment. The samples were characterized by means of scanning electron microscopy techniques, energy dispersive X-ray analysis and differential scanning calorimetry. It was shown that electric pulse treatment with an exposure time of less than 1 second leads to a significant change in the formed crystalline structure in comparison with the structure obtained by isothermal treatment. The microstructure of such samples in cross section is characterized by an uneven distribution of crystals over the thickness of the ribbon: columnar crystals are observed from the contact and non-contact sides of the ribbon, and large crystals are present in the inner part of the ribbon.

1. Introduction

The modern development of materials science places ever-higher demands on functional materials. One of the requirements is the ability of materials to show reactions to changes in external conditions and adapt to them (smart materials). Among such materials, materials with the shape memory effect (SME) are a striking example. Among such materials, TiNi-based alloys can be distinguished [1-4]. Such alloys are able to restore their shape when heated above the temperature of the austenitic transformation after deformation up to 8% [4-6]. These unique characteristics have provided widespread use of such materials. Fields of application of alloys with a shape memory effect are diverse and constantly expanding, these include: power engineering, instrumentation, robotics, microelectromechanical systems (MEMS), medicine, etc. [3-10]. Recently, alloys with the shape memory effect have demonstrated their effectiveness as micro dimensional drive devices [10-13].
Often, such devices require instantaneous tripping at temperatures close to room temperature, as well as a minimum hysteresis of the properties that characterize martensitic transformations (MT). One of the promising materials is the Ti50Ni25Cu25 alloy, in which the conversion hysteresis is several degrees in the temperature range slightly above room temperature [15-19]. In applications of micromechanical drives, reversible movements are often required. To obtain such properties in alloys with a shape memory effect, the two-way shape memory effect (TWSME) is used. Obtaining the effect of reversible shape memory in SME alloys is achieved by applying a counter-force to the alloy or by forming elastic stress fields in the alloy due to thermomechanical training [20, 21].

The formation of TWSME require special techniques for obtaining and processing materials. One of the interesting techniques for obtaining SME in thin ribbons from Ti50Ni25Cu25 alloy is the formation of bimorph structures. Such structures can be obtained by treating materials with concentrated energy sources [22]. A promising method for producing thin bimorph structures is ultrafast melt quenching. In this method, depending on the melt cooling rate determined by the technological parameters of the quenching process, an amorphous, crystalline, or amorphous-crystalline state can form in the ribs [21, 22]. With respect to the Ti50Ni25Cu25 alloy, when forming a thin ribbon by spinning or planar casting at melt cooling rates above 10⁶ °C/s, the entire volume of the ribbon is in an amorphous state, and at melt cooling rates below 10⁶ °C/s in the ribbon on a surface that is not contact with respect to the quenching wheel a surface crystalline layer is formed. Such a layered amorphous-crystalline material in the form of a thin ribbon is capable of exhibiting TWSME bending during thermal cycling in the range of martensitic transformation [22].

The first experiments made it possible to obtain a rapid-quenched amorphous-crystalline composite, to investigate the effect of reversible shape memory, which it showed, and to develop micro-grips for various purposes on its basis [10, 22]. Such a new unique material with TWSME has the advantages of simplicity and manufacturability of obtaining this effect in such alloys in comparison with the known processing methods, which often cannot be applied to fine materials. For the further development of technologies for obtaining a unique TWSME, it becomes relevant to study the effect of thermomechanical processing of rapidly quenched amorphous-crystalline composites on the physicomechanical properties and the inherent effects of reversible shape memory. In order to obtain new properties and modify existing effects of shape memory, additional heat treatment logically begs. One of the interesting heat treatments is the processing by a short pulse of electric current, which allows heating the sample in a short time [23]. An interesting fundamental problem is to study the basic laws of structure formation and the formation of physicomechanical properties in a rapidly-quenched layered amorphous-crystalline composite based on a TiNiCu alloy during isothermal and electric pulse treatment processing, and also to develop methods for creating new structural-composite functional materials with TWSME for micromechanical devices.

The aim of this work is to study the effect of electric pulse and isothermal treatment of layered amorphous-crystalline Ti50Ni25Cu25 alloys on their structure and thermomechanical properties.

2. Experimental part
For the study was chosen alloy Ti50Ni25Cu25 (at.%). The alloy of this composition is characterized by a sufficiently high tendency to amorphization and in the future allows you to obtain materials that have a shape memory effect with a narrow hysteresis of martensitic transformation.

In this paper, we studied samples of thin ribbons obtained by rapid-quenching technique (the method of spinning the melt), which consists in casting a jet of melt on the cylindrical surface of a copper wheel. The alloy ingots were prepared from ultrapure metals with sixfold remelting in an arc furnace in an argon atmosphere. The obtained blanks were melted in a quartz crucible in a helium atmosphere and extruded through a narrow nozzle in a crucible onto the surface of a rapid rotating copper wheel. In the process of obtaining the studied samples of ribbons by rapid-quenching technique, the cooling rate of the melt varied in the range from 10⁵ to 10⁶ °C/s, the width of the resulting ribbons - from 1 to 2 mm with a thickness of 30–50 µm.
The alloy samples were subjected to electric pulse treatment by passing a single pulse of electric current through the sample with a variable duration. In accordance with the calculations, the pulse amplitude at its specified duration was set in such a way as to ensure the release of thermal energy necessary to heat the sample to the crystallization temperature.

To provide the heat energy required for heating up the sample up to crystallization temperature, the relationship between the current value I and the duration $\Delta t$ of the electric pulse was calculated from:

$$I(\Delta t) = \frac{S}{\sqrt{\Delta t}} \times \sqrt{\frac{C \times \Delta T \times \rho_v}{\rho}}$$

where $\rho_v$ — density of the amorphous ribbon, $\rho$ — electrical resistivity of the amorphous ribbon, $C$ — thermal capacity of the amorphous ribbon, $S$ — cross-section area of the ribbon, and $\Delta T$ — temperature interval from ambient temperature to crystallization one.

Samples were obtained with an electric pulse treatment time of 5; 1; 0.1; 0.01 and 0.001 seconds. For comparison, samples of the alloy were obtained, crystallized by standard isothermal heat treatment in a furnace at 500 °C with a holding time of 300 seconds.

The microstructures of the samples’ surfaces and their cross sections were studied using scanning electron microscope (SEM) «FEI Quanta 600 FEG». The metallographic cross-sections of the rapid-quenched ribbons were made using «Buehler» equipment: an «Isomet 1000» precision cutting machine, a «Simplimet 1000» automatic hot-pressing hydraulic press machine, and an «EcoMet 250+AutoMet 250» grinding and polishing machine. For additional revealing the structure, we used etching of the polished surface with a solution of HF(5%)+H2NO3(25%)+H2O(70%), followed by wiping with a 9% solution of H2O2.

The structure of the alloys was examined with a «PANalytical Empyrean» X-ray diffractometer using Cu-K$\alpha$ radiation, the Bragg–Brentano focusing scheme, and a hybrid monochromator.

The critical temperatures of martensitic transformation and crystallization of alloys from the amorphous state were determined by differential scanning calorimetry (DSC) with an «STA 449 F1 Jupiter» under the following conditions: temperature range from 25 to 500 °C, heating rate 10 °C/min, in a dynamic argon atmosphere. When studying the parameters of MT, heating and cooling cycles were performed in the range from 25 to 120 °C at a heating rate 5 °C/min in a dynamic argon atmosphere. This interval was chosen based on previous studies of MT in such alloys.

3. Results and discussion

In the work, from a series of obtained samples of rapid-quenched ribbons for research, we selected a ribbon obtained at an alloy cooling rate of about 8.5·10$^5$ °C/s. The formed rapid-quenched ribbon has the necessary requirements for uniformity of geometric characteristics, 28-30 microns thick and about 1.5 mm wide. X-ray diffraction studies have confirmed that in this ribbon on the non-contact surface relative to the quenching disk there is a thin crystalline layer on the main amorphous part of the ribbon (from the contact surface side).

The study of the cross section of the rapid-hardened ribbon allowed us to characterize the structure of the ribbon. The cross-section view of the Ti$\text{50Ni}_{25}\text{Cu}_{25}$ alloy ribbon under study is shown in figure 1. From the data obtained, it is clear that the surface contains a crystalline surface layer with a thickness of 2.5 to 3 μm and a grain size in the cross section of 1 to 2.5 μm (Figure 1). The surface crystalline layer has a predominantly uniform thickness with slight thinning at the edges of the ribbon, and the amorphous part is characterized by the predominant absence of crystalline inclusions in the amorphous matrix. Etching of the ribbon to identify crystal structures on the surface of the ribbon slot in its amorphous part leaves a plaque, the removal of which leads to a strong etching of the crystal layer.
Figure 1. SEM images of the characteristic cross-section of the layered amorphous-crystalline ribbons of the Ti$_{50}$Ni$_{25}$Cu$_{25}$ alloy (a), after etching (b, c).

The crystallization processes in the obtained amorphous-crystalline ribbon of the Ti$_{50}$Ni$_{25}$Cu$_{25}$ alloy were studied by differential scanning calorimetry in the temperature range from 25 to 500 °C with a heating rate of 10 °C/min in a dynamic argon atmosphere (Figure 2). In the obtained curve, in the temperature range from 435 to 465 °C, there is a two-stage peak of heat generation responsible for the crystallization process. The first stage of the crystallization process of a layered amorphous crystalline sample is extended (flat), it occurs in the range from 435 to 450 °C. Such an observed gentle beginning of the crystallization process is obviously a consequence of the presence of a surface crystalline layer in the investigated ribbon. The boundary of the crystalline layer with the amorphous part is a region of stresses, a center of nucleation (nucleation) and crystal growth. The pronounced process of devitrification of the amorphous part is not observed, since this process intersects in the first stage. The second stage of the crystallization process is observed in the range from 450 to 465 °C; the corresponding peak of heat release is close to crystallization from the amorphous state typical of a given alloy. The temperature of the crystallization peak is 455 °C, the crystallization energy is 27.4 J/g.
Based on the obtained DSC data, heat treatment of amorphous-crystalline ribbons of the Ti$_{50}$Ni$_{25}$Cu$_{25}$ alloy was carried out by the methods of electric pulse and isothermal heat treatment. A series of samples was obtained from the initial amorphous-crystalline ribbon with an electric pulse heat treatment duration of 5; 1; 0.1; 0.01; 0.001 seconds. Also, to compare the properties, according to the standard method, a sample was obtained isothermally crystallized in an oven for 300 seconds at a temperature of 500 °C.

Samples of the processed Ti$_{50}$Ni$_{25}$Cu$_{25}$ alloy ribbons were characterized by X-ray diffraction analysis. The data of X-ray diffraction studies obtained from the contact and non-contact surfaces of the ribbons are shown in Figure 3. From the obtained data it is seen that the structure of rhombic martensite B19 is observed in all processed (crystallized) samples, while in the initial amorphous-crystalline ribbon on a non-contact surface together with the martensitic phase, there is a residual austenitic phase B2, apparently due to mechanical stresses in the surface crystalline layer. During heat treatment, relaxation of mechanical stresses occurs in the surface crystalline layer, at which the austenitic phase B2 passes into the martensitic B19. No peaks from crystalline phases are observed from the non-contact surface in the initial amorphous-crystalline ribbon, the layer is amorphous.

In an amorphous-crystalline ribbon after isothermal crystallization, as well as in the state after quenching (initial) from a non-contact surface, a non-standard arrangement of the main reflexes of the B19 type structure is observed in the range of 58-65 degrees, and peaks with lower intensity are located in the range of 28-32 degrees. Reflections from phase B2, observed in the initial state, are absent. X-ray diffraction studies from the contact side of the ribbon showed the presence of the main reflections of the B19 phase located in the region of 38-46 degrees that are standard for this ribbon of alloy. It should be noted that the intensity of the peaks from the contact surface is much lower than from the non-contact surface. This difference is explained by the strong texturing of the crystalline layer from a non-contact surface. This characteristic was shown in studies on similar materials with a thicker surface crystalline layer [21, 22]. From the data obtained it follows that the initial texturing of the surface crystalline layer is preserved. Thus, with isothermal crystallization of a layered amorphous crystalline ribbon from the contact and non-contact sides, structures with different crystallographic orientations are formed.
After electric pulse heat treatment (dynamic crystallization) of the amorphous-crystalline ribbon of the Ti₅₀Ni₂₅Cu₂₅ alloy, the crystallographic reflections from the non-contact surface have a similar character as after isothermal processing, the non-standard arrangement of the main reflexes of the B19 type structure is repeated. Crystallographic reflections from the contact side of the ribbon after electric pulse heat treatment are similar to the reflections obtained after isothermal treatment. Reflections of the B19 phase are observed, located in the region of 38-46 degrees (main), and less pronounced reflexes in the region of 58-65 degrees. Reflexes located in the region of 28-32 degrees, as in the case of isothermal treatment, are absent (therefore, this range is not indicated).

To study the micro- and macrostructure in the cross section of a ribbon isothermally crystallized in a furnace and a series of ribbons that underwent electric pulse heat treatment, their cross sections were made.

Figure 4 shows SEM images of the structure of a sample crystallized in an oven for 300 seconds at a temperature of 500 °C and a sample crystallized by an electric current pulse of 5 seconds duration.

**Figure 3.** X-ray diffraction patterns of the contact (a) and non-contact (b) surface of the Ti₅₀Ni₂₅Cu₂₅ rapid-quenched ribbons in initial, electric-pulse heat treatment and furnace isothermal treatment states.

![X-ray diffraction patterns](image)

**Figure 4.** SEM images of the cross-sectional structure of Ti₅₀Ni₂₅Cu₂₅ alloy ribbons after isothermal treatment at 500°C for 300 seconds (a) and an electric pulse heat treatment during 5 seconds (b).

![SEM images](image)

It is established that the isothermal treatment of the amorphous crystalline ribbon leads to the formation of a bimorphic crystalline structure in the ribbon, consisting of a recrystallized crystalline layer (from the non-contact side) and a crystalline layer formed from an amorphous matrix (from the part from the contact side). Formed structures have a clearly defined interface. The non-contact crystallized crystalline layer is characterized by an average thickness of 5-6 microns, note that the
initial thickness of the crystalline layer before heat treatment was 2.5-3 microns. The data obtained indicate the growth of the crystalline layer during isothermal processing. The crystalline layer formed from the amorphous part on the contact side is characterized by a uniform structure with an average crystal size of 0.5-1.5 \( \mu \text{m} \).

When exposed to an amorphous-crystalline ribbon by an electric current pulse lasting 5 seconds, a structure is formed that is close to the structure obtained by isothermal crystallization, but with slightly smaller grains and a smaller thickness of the crystalline layer.

When an amorphous-crystalline ribbon is exposed to an electric current pulse with a current duration of less than 1 second, significant changes in crystal formation are observed in the sample as a whole and in the amorphous layer in particular (Figures 5 and 6). The main one is that in dynamically crystallized alloys there is an inhomogeneous distribution of crystals over the thickness of the ribbon: a columnar structure of crystals is observed near the surfaces of the ribbon, and single or grouped larger crystals are present in the bulk of the ribbon. In the cross-sectional structure of the ribbon sample heat-treated by dynamic crystallization, the boundary of the initial crystalline layer is not observed in crystals from a non-contact surface. This fact indicates the growth of observed crystals from a non-contact surface from the initial size of the surface crystalline layer.

When an amorphous-crystalline ribbon is exposed to an electric current pulse of 1 second duration, columnar crystals are observed from the contact and non-contact sides of the ribbon. Large crystals with characteristic sizes of 5–10 \( \mu \text{m} \) are present in the bulk of the ribbon. The thickness of the layer of columnar crystals on the non-contact side is noticeably larger (on average 9 \( \mu \text{m} \)) than the thickness of the layer of columnar crystals on the contact side (average 6 \( \mu \text{m} \)). The sizes of columnar crystals across are on the non-contact side noticeably larger than on the contact side.

![Figure 5. SEM images of the cross-sectional structure of Ti50Ni25Cu25 alloy ribbons after an electric pulse heat treatment during 1 second (a) and 0.1 second (b).](image)

When an amorphous-crystalline ribbon is exposed to an electric current pulse of 0.1 second duration, columnar crystals grow, grow towards each other, and large crystals grow larger (Figure 5 b). The thicknesses of the layers of columnar crystals from the non-contact and contact sides are aligned (approximately 9-10 microns), their dimensions across the non-contact side are more than about two times (4-5 microns) from the contact side.

When exposed to an amorphous-crystalline ribbon by an electric current pulse of 0.01 second duration, columnar crystals from the surface grow deeper into the ribbon to crystals formed in the bulk of the ribbon (Figure 6 a, b). A part of the columnar crystals is in contact in the center of the ribbon, and a uniform boundary is formed.
Figure 6. SEM images of the cross-sectional structure of Ti50Ni25Cu25 alloy ribbons after an electric pulse heat treatment during 0.01 second (a, b) and 0.001 second (c, d).

When an amorphous-crystalline ribbon is exposed to an electric current pulse of 0.001 second duration in the volume of the amorphous part of the ribbon, large crystals break up into fractions, and columnar crystals from the contact side of the ribbon, in some areas, touch the center of columnar crystals on the non-contact side of the ribbon (Figure 6 c, d). The fraction of crystals formed from the volume of the amorphous part of the ribbon decreases, and their shape takes the form of lenses. The crystals on the non-contact side of the ribbon are thinner than the previous samples, with characteristic sizes 1-2 microns across.

The obtained SEM cross-sectional data confirm the patterns observed in the X-ray diffraction analysis. On the obtained structures (Figures 4-6), it is confirmed that during the heat treatment of a layered amorphous-crystalline ribbon from the contact and non-contact sides, structures with different crystallographic orientations are formed.

The study of the processes of martensitic transformations (MT) flow in the processed samples of the amorphous-crystalline ribbon of the Ti50Ni25Cu25 alloy was carried out by differential scanning calorimetry in heating and cooling cycles in the range from 25 to 120 °C at a rate of 5 °C/min (Figure 7). This interval is selected on the basis of previous studies of MT in such alloys [16, 23].
The DSC curves obtained show that in the initial sample, no transformations occur during heating and cooling. A shift in the MT temperatures is observed in the sample with the action of an electric current pulse for 5 seconds. This sample is a transition between samples with an electric pulse exposure time with durations of 5 seconds and 0.1 seconds. The presence of peak broadening and two-stage process of phase transformations is especially pronounced in the sample with an electric pulse exposure time of 1 second, which is consistent with the SEM data and is explained by a different grain size distribution in the structure, which is characterized by large values of the enthalpy of the magnetic field. In samples with electric pulse exposure lasting 0.1-0.001 seconds, a less pronounced two-stage transformation and the same range of MT intervals are observed. Table 1 shows the critical temperatures of the beginning and end of the magnetic field, as well as the enthalpy of transformations.

| Time   | $M_s$ °C | $M_f$ °C | $A_s$ °C | $A_f$ °C | $\Delta H_M$, J/g | $\Delta H_A$, J/g |
|--------|----------|----------|----------|----------|-------------------|-------------------|
| 300 s  | 64.6     | 58.0     | 65.6     | 71.3     | 13.2              | -12.7             |
| 5 s    | 59.0     | 54.2     | 61.8     | 67.9     | 13.1              | -12.7             |
| 1 s    | 69.2     | 58.9     | 65.4     | 73.6     | 14.4              | -13.1             |
| 100 ms | 66.1     | 57.2     | 63.7     | 70.9     | 12.6              | -12.5             |
| 10 ms  | 66.0     | 58.9     | 65.8     | 71.2     | 13.8              | -12.8             |
| 1 ms   | 65.3     | 58.1     | 65.7     | 71.0     | 13.1              | -12.3             |

A study of the manifestations of shape memory effects inherent in the Ti$_{50}$Ni$_{25}$Cu$_{25}$ alloy was carried out by the method of bending deformations. The study showed the presence of pronounced SME in all heat-treated samples. Moreover, in the samples of amorphous-crystalline ribbons heat-treated with an electric current pulse, a weak effect of reversible shape memory is observed, which is realized by bending the ribbon during its thermal cycling in the MT range.

4. Conclusions

Electric pulse crystallization of an amorphous crystalline rapid-quenched Ti$_{50}$Ni$_{25}$Cu$_{25}$ alloy leads to the formation of new structural states.

It was found that electric pulse treatment with an exposure time of less than 1 second leads to a significant change in the formed crystalline structure in comparison with the structure obtained by
isothermal treatment. The microstructure of such samples in cross section is characterized by an uneven distribution of crystals over the thickness of the ribbon: columnar crystals are observed from the contact and non-contact sides of the ribbon, and large crystals are present in the inner part of the ribbon.

A decrease in the time of electric pulse exposure to 0.01 and 0.001 seconds leads to an increase in the fraction of columnar crystals, while a decrease in structural elements is observed - columnar crystals from the contact surface are thinned, and the crystals are crushed in the volume of the ribbon. It was found that columnar crystals from the surface go deep into the ribbon to crystals formed in the bulk. Areas were discovered in which columnar crystals are in contact with each other in the inner part of the ribbon, thus creating a uniform boundary. The structure of columnar crystals on the noncontact side retains the texture of the initial surface crystalline layer.

Acknowledgements
The study was supported by the Russian Science Foundation (project no. 19-72-00145).

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