Thermoelastic Properties and Elastocaloric Effect in Rapidly Quenched Ribbons of Ti$_2$NiCu Alloy in the Amorphous and Crystalline State

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Abstract: The thermoelastic properties and the elastocaloric effect (ECE) were studied in rapidly quenched ribbons of the Ti$_2$NiCu alloy samples in amorphous and crystalline states under periodic mechanical tension with a frequency of up to 50 Hz. In the amorphous samples, elastic behavior is observed, described by Hooke’s law, with a high coefficient of thermal expansion $\alpha = 1.7 \times 10^{-4}$ K$^{-1}$. Polycrystalline ribbons of the Ti$_2$NiCu alloy have the classical shape memory effect (SME), the temperatures of the forward and reverse thermoelastic martensitic transitions being $M_s = 345$ K, $M_f = 325$ K, $A_s = 332$ K, and $A_f = 347$ K and the coefficient of the dependence of the transition temperature on mechanical stress being $\beta = 0.12$ K/MPa. The experimentally measured value of the adiabatic temperature change under the action of mechanical stress (ECE) in the amorphous state of the alloy at room temperature ($T_r = 300$ K) was $\Delta T = -2$ K, with a relative elongation of $\varepsilon = 1.5\%$ and a mechanical stress of $\sigma = 243$ MPa. For crystalline samples of Ti$_2$NiCu alloy ribbons, the ECE is maximum near the completion temperature of the reverse thermoelastic martensitic transformation $A_f$, and its value was 21 K and 7 K under cyclic mechanical loads of 300 and 100 MPa, respectively. It is shown that the ECE value does not depend on the frequency of external action in the range from 0 to 50 Hz. The specific power of the rapidly quenched ribbon was evaluated as a converter of thermal energy at an external mechanical stress of 100 MPa; its value was 175 W/g at a frequency of 50 Hz. The thermodynamic model based on the Landau theory of phase transitions well explains the properties of both amorphous ribbons (reverse ECE) and alloy ribbons with EPF (direct ECE).

Keywords: functional alloys; amorphous alloys; Ti$_2$NiCu; shape memory effect; thermoelastic martensitic transition; elastocaloric effect; phase transition kinetics; specific power of energy conversion

1. Introduction

In recent years, the new solid-state functional materials and their properties have attracted great attention. They have found important applications in sensor and actuator technology. For example, intermetallic alloys with a shape memory effect (SME), which is caused by thermoelastic martensitic transition occurring in the solid phase of the alloy, have a remarkable property to restore their shape under significant deformation and perform mechanical work under the influence of temperature or magnetic fields [1]. At the end of the 20th century, the work [2] played an important role in the development of caloric effects. It increased the interest of the world scientific community in their study. In this work, a “giant” elastocaloric effect (ECE) during a phase transition in a solid-state alloy Fe$_{49}$Rh$_{51}$ is studied in a magnetic field and under tensile stress. In recent years, proposals have been made on the use of solid-state functional materials in alternative energy. To this end, new materials are being studied that demonstrate various caloric effects, such as magnetocaloric, elastocaloric, and electrocaloric, arising in samples of solid-state materials under the influence of external fields. The essence of the above effects is a strong change...
in the entropy and temperature of these materials under the influence of external fields: magnetic, electric, and mechanical stress fields, which is maximum, respectively, near the phase transitions affecting the corresponding order parameters: magnetic moment, electric polarization, or structural order parameters.

The ECE, that is, a change in the temperature of solids during adiabatic deformation, has been known for a long time [3]. Recently, a lot of work has been devoted to the study of ECE [4–13]. It was shown in [4] that Ti–Ni alloy with a thermoelastic martensitic transition and SME has a significant ECE (more than 20 K). This is not surprising, since ECE is, in fact, the inverse SME effect, and the Ti–Ni alloy is a recognized record holder for SME parameters among functional intermetallic compounds. However, in [5–7], it was noted that, despite the giant ECE, this alloy actively accumulates fatigue defects, which reduce its value with repeated periodic loading. The authors of [4] proposed the study of ECE in other materials, such as intermetallic compounds with SME: Ni$_2$FeGa, Ni$_2$TiCu, CuZnAl, and CoNiAl, which are also recognized as promising for elastocaloric cooling due to their high thermomechanical characteristics. A colossal value of the ECE was recorded in the Ni–Mn–Ti alloy, up to 31.5 K [9]. The disadvantages of this and other works include measurements of the ECE at a single deformation or at low frequencies (of the order or less than 1 Hz). As a result, the important questions about the maximal attainable energy flow rate induced by mechanical stress, at the thermoelastic martensitic transition, and the ultimate specific power of thermal energy conversion by a working fluid based on a solid-state material with ECE remains unanswered.

The rapidly quenched Ti$_2$NiCu alloy, which is obtained in the form of ribbons by the spinning method, attracts much attention due to its manufacturability and high values of the functional properties of SME [14–17]. The thermoelastic martensitic transition and SME in this alloy were demonstrated on nanoscale size of samples [18], and composites based on it were found to be very convenient for creating actuators with reversible thermoinduced deformation, which are recorded in miniature size and frequency performance [19]. It was proposed to use composite nano-instruments with SME for technologies of the bot. 73 tom-up to nanomanipulation and nano-assembling [20].

The ECE under tensile stress in the Ti$_2$NiCu alloy was very convenient to be studied, since the alloy was obtained in the form of thin ribbons with a thickness of the order of 30–40 µm with high strength, up to hundreds of MPa, and a convenient temperature range of the martensitic phase transition in the range of 40–60 °C. In addition, the Ti$_2$NiCu alloy obtained by ultrafast quenching on a rotating copper wheel immediately after preparation turned out to be amorphous or layered amorphous-crystalline. The presence of alloy samples in a polycrystalline state, in which a thermoelastic martensitic transition of the 1st order and SME were present, and in amorphous state in which there was no long-range crystalline order and, therefore, a thermoelastic phase transition was not observed, makes this alloy very convenient for a comparative study of the role of the phase transition to nature SME and ECE.

The aim of this work is the experimental and theoretical study of the thermoelastic properties and ECE in the rapidly quenched ribbons of the Ti$_2$NiCu alloy in amorphous and crystalline states at different values of the frequency of the external tensile stress. Additionally, the aim is to estimate the maximum specific power of heat conversion by a working body made of a rapidly quenched Ti$_2$NiCu ribbon.

2. Materials and Methods

Samples of the Ti$_2$NiCu alloy were obtained by ultrafast quenching at a speed of $10^7$ K/s in the form of a ribbon with 30–40 µm thickness and 1.5 mm wide. After quenching, the alloy was mainly in the amorphous state [14].

To study the thermoelastic properties and ECE, we selected samples of rapidly quenched ribbons of Ti$_2$NiCu alloy approximately 10–12 cm long, 1.5 mm wide, and 36 µm thick [21]. Afterwards, rapid quenching samples were in amorphous state. Ribbons were annealed at $T = 450$ °C for 2 h with cooling in air to gain a crystalline state and study
the SME and ECE. Figure 1 shows the results of study of the structural phase transition by differential scanning calorimetry (DSC). The characteristic temperatures of the start and finish of the direct and reverse martensitic transitions of the samples were $M_s = 62.0 \, ^\circ C$, $M_f = 51.1 \, ^\circ C$, $A_s = 59.8 \, ^\circ C$, and $A_f = 67.5 \, ^\circ C$, and the latent phase transition energy was $\lambda = 11.5 \, J/g$.

![Figure 1. DSC data (blue line—cooling, red line—heating) for rapidly quenched Ti$_2$NiCu ribbon annealed for 2 h at a temperature of 450 °C.](image)

The setup described in [22] was used to determine the thermomechanical properties. The principle of operation of the experimental setup is based on the stretching of an alloy specimen of an alloy with SME under constant load at a variable temperature.

An original experimental setup was constructed to study the ECE in rapidly quenched Ti$_2$NiCu alloy ribbons at a frequency up to 50 Hz. It consisted of the actuator (asynchronous electric motor with a crank mechanism and a spring) rigidly fixed to the platform and connected to a variable voltage source. The actuator provided periodic deformation of the test sample, rigidly fixed from one end, with the other end attached to the spring, which, in turn, was attached to the actuator connecting rod. The temperature of the sample was changed by passing an electric current through it. A sweep current generator was used as a source, providing gradual heating and cooling of the ribbon through the martensitic transition range, with a peak current value of about 1 A. The surface temperature of the sample was measured remotely using an infrared camera brand Flir SC-7000 with a frame frequency of up to 400 Hz. To increase the accuracy of thermal measurements, the region of the ribbon on which the camera was focused was blackened. A personal computer was used to collect and process high speed thermograms recorded by camera and measured data.

3. Results

3.1. Thermoelastic Properties of Ti$_2$NiCu Melt Spun Ribbon

Figure 2 shows the dependencies of the deformation of the ribbon annealed at a temperature of 450 °C for 2 h under the action of different constant mechanical stresses.
Temperature dependences of sample deformation at various constant mechanical loads (indicated in the inset of the graph, MPa) for an annealed strip (up arrow—direct transformation upon cooling, down arrow—reverse transformation upon heating).

The amorphous ribbon sample did not exhibit a thermoelastic martensitic transformation, which was pronounced in the annealed sample (Figure 2). The maximum reversible deformation of the annealed ribbon sample was 2.5% at a load of 405 MPa.

Contrary to the amorphous ribbon, the polycrystalline ribbon, with an increase of temperature, was compressed in the vicinity of thermoelastic martensitic transition. At the higher external stretching load, thermal cycling was carried out at the higher compression. This behavior indicates a clear SME due to two-stage thermoelastic martensitic transformation in the temperature range 325–360 K, depending on the stress applied. The average value of the coefficient of dependence of the temperature Af of the end of the transformation from martensite to austenite on mechanical stress was found: \( \beta = 0.12 \, \text{K/MPa} \). The calculated value of the coefficient of thermal expansion for the annealed ribbon in the low temperature martensitic state well below Mf was approximately \( 4.2 \times 10^{-7} \, \text{K}^{-1} \).

3.2. ECE in Rapidly Quenched Ribbons

3.2.1. ECE in Amorphous Rapidly Quenched Ti2NiCuRibbons

As a result of the measurements of ECE, IR thermograms were obtained and processed. An example of images obtained by a high-speed IR camera from an unstretched and stretched amorphous ribbon of a Ti2NiCu alloy is shown in Figure 3a,b. When stretched, the tape cools were reflected by the color change of the image elements (pixels). For quantitative measurement of the ECE, temperature distribution diagrams of the number of pixels were constructed (Figure 3c,d). In each of the diagrams, a background peak with a room temperature of 300 K (shown in black) and a peak corresponding to the temperature of the sample are highlighted.

It can be seen that this peak moved to the region of lower temperatures during ribbon stretching deformation, which occurred due to the inverse ECE in the elastic ribbon. No pronounced effect of the change to the frequency of the external force cycles up to 10 Hz on the value of the ECE was observed, indicating that, in this range, the ECE does not depend on the frequency.
3.2.2. ECE in the Polycrystalline Ti$_2$NiCu Ribbons

Thermograms are shown on Figure 4, which shows the crystalline samples of Ti$_2$NiCu alloy ribbons. The thermograms record the spatial distribution of the sample temperature under periodic exposure to tensile stress with increasing and decreasing average ribbon temperature in the region of the martensitic transition. Photographs from the thermal imager screen of a strip sample in a stretched and undeformed state, which are shown in Figure 4a,b, respectively, demonstrate uneven heating and cooling during cyclic loads.

As a result of processing the thermograms, the dependences of the sample temperature on time during heating and back cooling were obtained. An example of such a dependence is shown in Figure 5 at a cyclic load with the amplitude of 100 MPa with an exposure frequency of 10 Hz. The rate of gradual heating of the sample (for measuring the ECE over the entire range of the phase transition and near it) is 4 K/s, and the rate of back cooling is 3.5 K/s. It was experimentally shown that, with a frequency of tension-compression cycles of more than 1 Hz, the process was adiabatic and external heating/cooling did not contribute to the ECE. As can be seen in Figure 5, at a periodic load with an amplitude of 100 MPa and a frequency of 10 Hz, and an increase and then a decrease of the current through the sample, the heating rate of the sample was 4 K/s and the maximum value of the ECE was 7 K.
Figure 4. Thermograms of a polycrystalline tape made of Ti$_2$NiCu alloy deformed at a frequency of 6 Hz: a—a tape under load, b—a tape without a load. Dependences of the sample temperature on time (c) and (d) at the time point corresponding to the recording of thermograms (a) and (b), respectively.

Figure 5. The dependence of the temperature of the sample on time during heating and cooling at a cycle frequency of the driving force of 10 Hz. The maximum load of 100 MPa.
Analyzing the thermograms obtained at different parameters of external influences on the Ti$_2$NiCu ribbons, it was found that the maximum value of the ECE at cyclic loads was 21 K (Figure 6) at a load of 300 MPa and the frequency of the loading cycles was 3 Hz.

Figure 6. The dependence of the temperature of the sample on time during gradual heating under the influence of external periodic mechanical stress with a frequency of 3 Hz, with a maximum value of 300 MPa.

During the experiments, it was shown that the value of the ECE did not depend on the frequency of the external driving force (Figure 7).

Figure 7. Dependence of ECE on the frequency of tensile and compression cycles of the sample at a maximum mechanical stress of 100 MPa at a temperature of Af.

To assess the possibility of using rapidly quenched Ti$_2$NiCu ribbons as a working fluid in elastocaloric heat pumps, in Figure 8, the plot of the specific power versus the frequency of the external periodic force on the studied tapes is given.

To get the formula of the specific power, we use the relation of the thermal energy of the sample:

\[ Q = c_M \cdot m \cdot \Delta T \]  

where $c_M$ is the specific heat of the sample in martensitic state, since the ECE is due to the induction of overheated martensite under the action of an external load, $m$ is the mass of the sample, and $\Delta T$ is the change in the temperature of the sample (ECE).
Figure 7. Dependence of ECE on the frequency of tensile and compression cycles of the sample at a maximum mechanical stress of 100 MPa at a temperature of Af.

To assess the possibility of using rapidly quenched Ti$_2$NiCu ribbons as a working fluid in elastocaloric heat pumps, in Figure 8, the plot of the specific power versus the frequency of the external periodic force on the studied tapes is given.

Figure 8. Dependence of the specific power of a working fluid based on a Ti$_2$NiCu polycrystalline ribbon on the frequency of external mechanical stress.

By definition, the specific power (power per 1 g of mass) for one period of oscillation ($\tau$) is:

$$W = \frac{Q}{m \cdot \tau}$$

(2)

Thus, substituting (1) expression into (2) and taking into account that $\tau = 1/f$ ($f$—frequency of oscillations), we obtain:

$$W = c_M \cdot f \cdot \Delta T$$

(3)

where $c_M$ is the specific heat of the sample in martensitic state, $f$ is the frequency of cycles of exposure to external mechanical stresses, and $\Delta T$ is the ECE. At $f = 50$ Hz (the maximum frequency provided by the experimental setup) and a mechanical stress of 100 MPa ($\Delta T = 7$ K), we obtain $W = 175$ W/g.

4. Discussion

In this work, we studied the ECE in rapidly quenched ribbons of the Ti$_2$NiCu alloy in various structural states. ECE in an amorphous and polycrystalline alloy at a temperature corresponding to the temperature Af of the polycrystalline Ti$_2$NiCu alloy has a different sign, which is in qualitative agreement with estimations that were done based on the measurement data of their thermoelastic properties.

The very derivation of the formula for calculating the dependence of deformation on temperature in classical solids is theoretically described in [19], using the expansion of the Helmholtz free energy function to the second order in deflation and temperature. The calculated ECE value of the amorphous Ti$_2$NiCu alloy ($\Delta T = 2$K) can be obtained from the equation:

$$\Delta T = -\frac{\alpha K}{c_p} \frac{\Delta l}{l}$$

(4)

where $\Delta T$ is the ECE value, $T$ is the initial temperature of the sample ($\Delta T << T$), $\alpha$ is the coefficient of thermal expansion, $K$ is the coefficient of volume elasticity ($K = E/(3 (1-2 \nu))$, $E$ is Young’s modulus, $\nu$ is Poisson’s ratio), $c$ is the specific heat of the sample (without martensitic transition), $\rho$ is the specific density of the sample, and $\Delta l/l$ is the relative strain of the sample under tension ($l$ is the length of the sample, $\Delta l$ is the linear deformation of the sample). It corresponds very well to the experimentally obtained value of the ECE.

In a polycrystalline alloy, the greatest effect is observed at a temperature Af. This is explained by the formation of a low temperature structural phase under the influence of
mechanical stress and, in article [9], is obtained and theoretically described in detail using
the Gibbs thermodynamic potential for materials with shape memory:

$$\Delta T(S, O \rightarrow f = p, \sigma) \simeq \frac{T \Delta S(T, O \rightarrow f = p, \sigma)}{c_M} \tag{5}$$

where T is the sample temperature, $\Delta S$ is the entropy change, and $c_M$ is the specific
heat of the sample in martensitic state. We substitute the values ($\lambda = 1.15 \times 10^4$ J/kg,
$c_M \approx 500$ J/(kg K)), and taking into account that $T \Delta S = \lambda$ (\lambda is the latent energy of phase
transition), we obtain $\Delta T \approx 20$ K. In the present work, the experimentally measured
maximum value of the ECE is $\Delta T = 21$ K. Given the measurement error, we can conclude
that the entire high temperature phase under the influence of a mechanical stress of 300 MPa
completely passes into the low temperature phase. It follows that, in the experiment, the
maximum possible value of the ECE for this alloy was achieved with the used methods of
sample preparation.

The obtained maximum value of the ECE is in good agreement with the theoretical
estimate of the ECE described in [9].

It is also shown that the ECE value does not depend on the frequency of the external
driving force. From this we can conclude that the limit speed of the phase transition has
not been reached. Dependence-specific power on frequency shows linear character.

5. Theory

A first-order structural phase transition takes place in the material with SME. Proceed-
ing from this, we can say that the dependence $\sigma(\varepsilon)$ is nonlinear [22].

For the theoretical description of the ECE at a structural phase transition, we use the
expansion of the Helmholtz free energy function up to the fourth order in deformation and
up to the second order in temperature.

$$F = \frac{a}{2} \varepsilon^2 + \frac{b}{3} \varepsilon^3 + \frac{c}{4} \varepsilon^4 - \frac{A}{2} \Delta T^2 \tag{6}$$

where $a = a_0 *(T - T_c)$; but b, c, and A are temperature-independent constants. $T_c$ is the
phase transition temperature.

A-priory:

$$dF = -pdV - SdT \tag{7}$$

Since the studied effect implies only uniaxial deformations in the phase transition
region, and considering that $\sigma = -p$, we obtain:

$$dF = \sigma \varepsilon - SdT \tag{8}$$

From (8), we can conclude:

$$\frac{\partial F}{\partial (\varepsilon)} = \sigma \tag{9}$$

$$\frac{\partial F}{\partial (\Delta T)} = -S \tag{10}$$

Substituting expression (6) into (9), we obtain the thermal equation of state (11). The
graph of this expression is sketched in Figure 9. The coefficients describing a cubic
relationship, are taken as follow: $a = 1.4 \times 10^{10}$ Pa, $b = -1.5 \times 10^{12}$ Pa and $c = 5.3 \times 10^{13}$ Pa.
This property possessed in alloys with SME is called superelasticity. The characteristic
points of maximum and minimum on the red curve are points of phase stability loss. The
curve between them has no physical meaning and will not be observed in the experiment.
The blue curve gives a qualitative image of the experimental dependence corresponding to
a first-order phase transition with specific hysteresis.

$$\sigma = a \cdot \varepsilon + b \cdot \varepsilon^2 + c \cdot \varepsilon^3 \tag{11}$$
For solids, in contrast to gas, the change in entropy occurs in the vicinity of the temperature point \( T \neq 0 \); therefore, the concept of entropy \( S \) in a solid makes sense as a change in entropy \( \Delta S \). Then (10) has a form:

\[
\frac{\partial F}{\partial (\Delta T)} = -\Delta S
\]  

(12)

Substituting expression (6) into (12), we obtain the caloric equation of the state:

\[
\Delta S = A \Delta T - \frac{1}{2} a_0 \varepsilon^2
\]  

(13)

Since deformation occurs under adiabatic conditions (\( \Delta S = 0 \)), we obtain:

\[
\Delta T = \frac{a_0 \varepsilon^2}{2A}
\]  

(14)

The resulting expression for the ECE (14) shows a quadratic dependence on deformation during the phase transition. To estimate the \( \Delta T \) value, we take

\[
a_0 = \frac{a}{T_c - T_c}, \quad A = \frac{\Delta \varepsilon^2}{T_c - T_c}
\]

Where \( a = 1.4 \times 10^{10} \) Pa; \( (T - T_c) = 5 \) K—half the width of the hysteresis, \( c_M = 500 \) J/(kg·K)—specific heat capacity in martensitic state; \( \rho = 7 \times 10^3 \) kg/m\(^3\)—density; \( T = A \) K—sample temperature. Then \( a_0 = 2.8 \times 10^9 \) Pa/K; \( \varepsilon = 1.2 \times 10^{-2} \); \( A = 10^4 \) J/(m\(^3\)·K\(^2\)). As a result \( \Delta T = 20.1 \) K, which is well confirmed by the experiment (Figure 6).

6. Conclusions

We conclude that a comparative study of amorphous and polycrystalline melt spun ribbons of Ti\(_2\)NiCu alloy clearly revealed the drastic effect of thermoelastic martensitic transition. The ECE in amorphous and polycrystalline alloy at a temperature corresponding to the temperature \( A \) of the polycrystalline Ti\(_2\)NiCu alloy has a different sign, which is in qualitative agreement with the measurement data of thermoelastic properties.

It is shown that the maximum value of the ECE is achieved at the point corresponding to the completion of the inverse thermoelastic martensitic phase transition \( T = 67.5 \) °C. The

![Figure 9](image-url)
maximum ECE value in the Ti₂NiCu alloy is 21 K at a load of 300 MPa and a frequency of deformation cycles of up to 3 Hz. The value of the ECE does not depend on the frequency of tensile cycles up to 50 Hz.

The maximum specific heat transfer power calculated for a elastocaloric heat pump was estimated as \( W = 175 \text{ W/g} \), with an applied external mechanical periodic stress of 100 MPa and a frequency of 50 Hz and ECE as high as 7 K.

Theoretical calculations, according to thermodynamical model of the ECE for amorphous ribbons, showed good agreement with experimental results. The formula for ECE for polycrystalline ribbons predicts the maximum value of the ECE near martensitic transition and monotonic dependence of the adiabatic temperature change versus tensile stress until complete transition martensite-austenite has also been experimentally verified and confirmed.

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