ISSN 1062-8738, Bulletin of the Russian Academy of Sciences: Physics, 2017, Vol. 81, No. 11, pp. 1304–1309. © Allerton Press, Inc., 2017.
Original Russian Text © Yu.N. Vjunenko, V.V. Kozhushko, A.E. Volkov, E.V. Chernyaeva, 2017, published in Izvestiya Rossiiskoi Akademii Nauk, Seriya Fizicheskaya, 2017, Vol. 81, No. 11, pp. 1451–1457.

Acoustic Emission during the Thermal Cycling of Titanium Nickelide under Conditions of Uneven Heating

Yu. N. Vjunenko[a], V. V. Kozhushko[b], A. E. Volkov[c], and E. V. Chernyaeva[c], *

[a]*OOO Optimikst Ltd., St. Petersburg, 195426 Russia*

[b]*Belyi Metal–Polymer Research Institute, Belarus National Academy of Sciences, Gomel, 246050 Belarus*

[c]*St. Petersburg State University, St. Petersburg, 198504 Russia*

**e-mail: ech312@yandex.ru, lena@smel.math.spbu.ru*

Abstract—The behavior of acoustic emission (AE) upon thermal cycling of the T–Ni alloy is studied. It is shown that AE can be used to assess working parts made of shape memory alloy.

DOI: 10.3103/S106287381711020X

INTRODUCTION

The engineering of mechanisms that operate on the basis of the shape memory effect requires the development of means for monitoring the status of load-bearing members. OOO Optimikst Ltd. (St. Petersburg, Russia) has designed and uses a device known as SheR in which the necessary forces are generated by "metallic muscles" made of Ti–Ni alloy [1]. The mechanical stresses in them are far from the limit values of the material's durability, so the muscles operate steadily for many years. However, studies by Yu.D. Kravchenko et al. [2] have shown that devices made of titanium nickelide can be destroyed if substantial forces are generated during the heating of the material. Of special importance in this regard are state-of-the-art procedures for diagnosing the state of the material, particularly acoustic emission (AE), which allows us to study both the energy [3] and frequency parameters of AE signals [4–6] and thus to use AE to assess the state of metal parts, including those that operate under conditions of cyclic stress.

Martensitic transformations in Ti–Ni alloy were shown in [7–9] to be accompanied by intense AE. The generation of acoustic signals in this case depended on the state of the material and the change in its characteristics. However, the above works studied AE under conditions of the even heating of a sample's surface. In industrial processes, machine parts made of shape memory (SM) materials operate under gradients of temperature and strain, oscillations in force, and chemical composition. The aim of this work was to study AE upon the thermal cycling of Ti–Ni alloy subjected to uneven heating in order to assess the applicability of AE to monitor the status of devices using the shape memory effect (SME) [10] and operating under real industrial conditions.

EXPERIMENTAL

Planar samples of Ti–50.6 wt % Ni alloy 64 × 10 × 2.5 mm in size were prepared for our studies. The temperature dependences of their internal friction and elastic characteristics were measured on a DMA Q800 dynamic mechanical analyzer (TA Instruments, United States) using plate-bending circuit fixed at two points. Room temperature samples were placed in a chamber whose temperature was raised to 25°C and stabilized for 1 min. The rate of temperature change was 0.75°C/min in the range of 25 to 120°C. The Young modulus and loss-angle tangents for seven frequencies were measured in the range of 0.1 to 100 Hz. The relationship between the measured parameters was derived from the complex representation $E = E' + jE''$ of the Young modulus, where the real part describes the elasticity of the material and the imaginary part is associated with the internal losses of friction in the material. These are expressed as $\tan\delta = \frac{E''}{E'}$, the value of which is proportional to the losses. Figure 1 shows the absolute magnitudes of modulus E' and $\tan\delta$.

We found that a phase transformation develops upon heating in the temperature range of 38 to 55°C (Fig. 1). There was a peak of dissipation characteristics, the amplitude of which fell as the frequency rose. The elastic characteristics under high-temperature conditions were 10% higher than the corresponding values of martensite. At frequencies below 5 Hz, the Young modulus displayed a minimum in the range of 38–55°C. The absolute magnitude of the modulus grew along with the measuring frequency.

In all our experiments to study AE, the unfixed end of the plate was heated using a candle flame (Fig. 2),

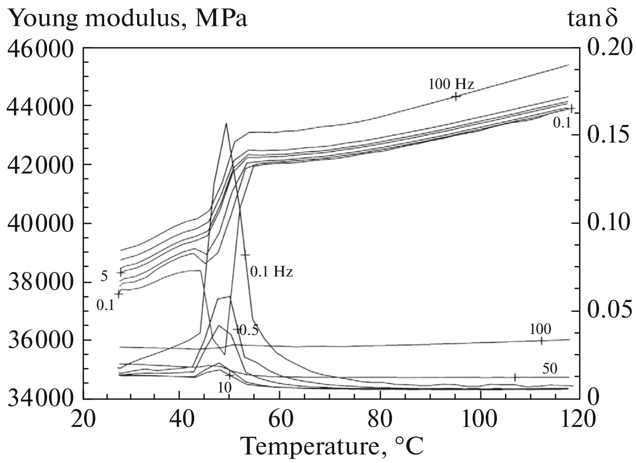

Fig. 1. Young modulus E' (upper curves) and tanδ (lower curves) as functions of temperature upon heating a sample made of the Ti–50.6 wt % Ni alloy at frequencies of 0.1, 0.5, 1, 5, 10, 50, and 100 Hz.

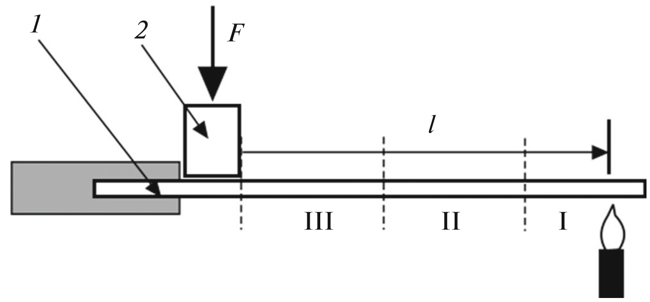

Fig. 2. Diagram of the device: (*1*) sample, (*2*) AE sensor; (*F*) clamping force, (*l*) distance from the AE sensor to the heating site; I–III, heating zones.

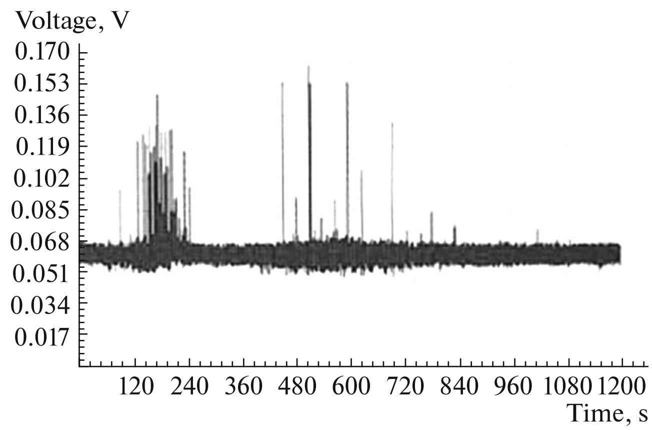

Fig. 3. Typical AE pattern in the heating–cooling cycle (heating with a candle flame in time intervals of 20 to 300 s from the start of an experiment; cooling at room temperature).

ensuring a temperature gradient over the length of the sample.

AE was recorded using a MSAE 1300WB-C sensor and a MSAE-FA010 amplifier with a total force of 80 dB. The sensor was installed on the top surface of the plate with clamping force $F \sim 2$ N. All AE signals recorded using the procedure for the digital recognition and analysis of spectral samples in [6] were divided into groups according to the shapes of the spectral density curves, and the spectral patterns (the averaged shape of the spectral power density curves) were analyzed for each group.

RESULTS AND DISCUSSION

Effect of Distance between the AE Sensor and the Heating Zone

The typical development of the AE during heating and cooling is shown in Fig. 3. The plate edge was heated in a time interval of 20 to 300 seconds from the beginning of each experiment. Signal generation upon heating (the reverse martensitic transformation in Ti–Ni) was noted in the time interval of 80 to 250 s. Depending on the experimental conditions, anywhere from several dozen to 1500 signals were recorded within this time.

Each sample was then cooled in air to room temperature. The most active AE was in this case observed in the time interval of 400 to 1000 s, after which occasional individual pulses appeared up to the end of recording, suggesting possible incompleteness of the direct transformation at room temperature.

Measurements showed that the AE depended strongly on length l from the heating zone to the sensor (see Fig. 2). When $l = 40$ mm, the first pulses were recorded at 160 seconds of heating; the total number of signals upon reverse transformation N_{heat} was 32. Moving the heater to distances of $l = 25$ and 10 mm accelerated the initial AE recording (i.e., shortened time t_0) and increased the number of AE signals upon heating (N_{heat}) and the total number of signals (N_{tot}) (Table 1).

The energy of the signals is shown as a function of time in Fig. 4, where only the main (most numerous) group is shown.

Our data led us to conclude that the AE signals generated by the reverse transformation were due to a phase transition in the bulk of the material near the sensor. Moving the source of heating closer to the sen-

Table 1

Heating zone	l, mm	t_0, s	N_{heat}	N_{tot}
I	40	160	32	114
II	25	100	122	959
III	10	60	340	1754

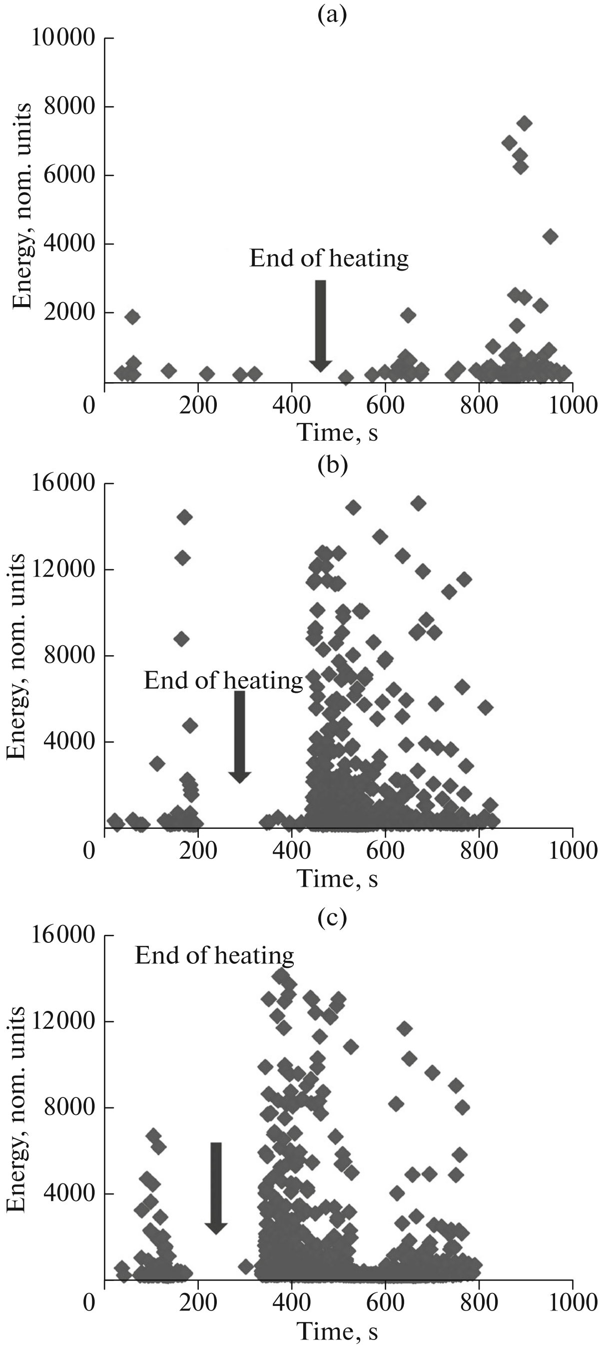

Fig. 4. Acoustic emission upon the heating of planar Ti–Ni samples in zones I–III (see Fig. 2) and cooling in air.

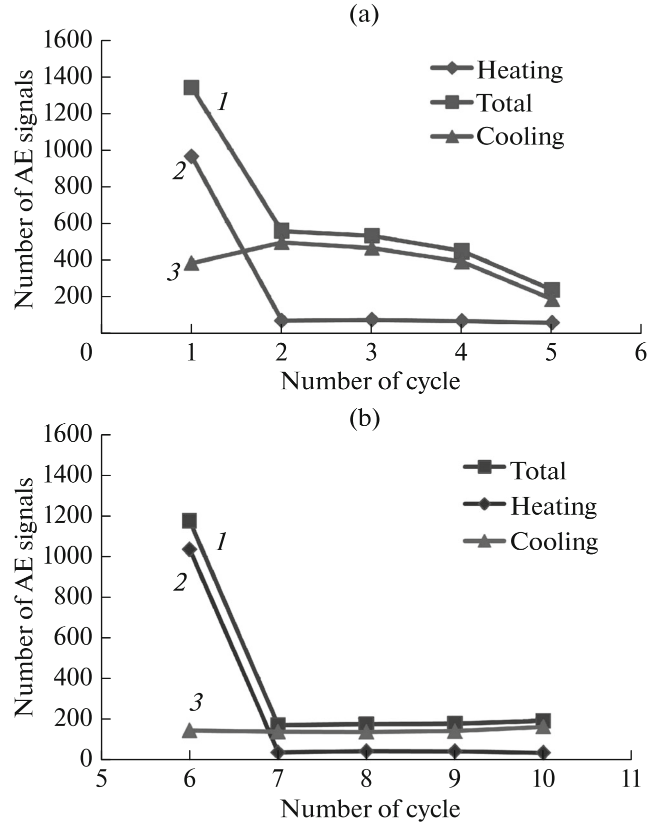

Fig. 5. (*1*) Total number of recorded AE signals and number of signals upon (*2*) heating and (*3*) cooling as a function of the number of thermal cycles: (a) 1–5 and (b) 6–10.

sor shortened the time required to warm the contact zone to the high-temperature state, and thus the time of initial AE recording. The interval of time in which the sample generated sound upon heating was reduced and the number and energy of signals grew. The total number of signals in the total heating–cooling cycle was also higher when the metal was heated near the sensor.

AE during Sequential Thermal Cycles

The considerable effect thermal cycling has on acoustic emission upon a material's transformation from martensite to austenite and *vice versa* was noted in [7]. Similar data were obtained for our samples annealed for 17 min at 500°C with subsequent water quenching. A total of two series of thermal cycles with five cycles in each series were performed, the samples being kept at room temperature for 48 h between the first and second series. The distance from the zone of heating to the sensor was 40 mm.

Figure 5 and Table 2 give the change in the number of recorded signals, depending on the number of the thermal cycle.

In the first series of thermal cycles (Fig. 5a), the total number of signals and the number of signals upon heating fell dramatically as early as in the second cycle. There was an increase in the number of signals in the second cycle upon cooling, but the number of signals in the following cycles fell gradually upon cooling.

The number of AE signals upon heating increased in the sixth cycle after keeping the samples at room temperature for 48 h (Fig. 5b). The number of signals

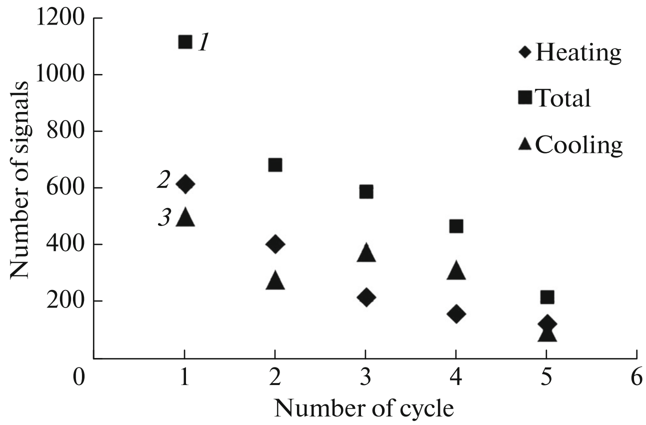

Fig. 6. (*1*) Total number of recorded AE signals and number of signals upon (*2*) heating and (*3*) cooling, as a function of the thermal cycle number in a sample cooled to −10°C prior to each thermal cycle.

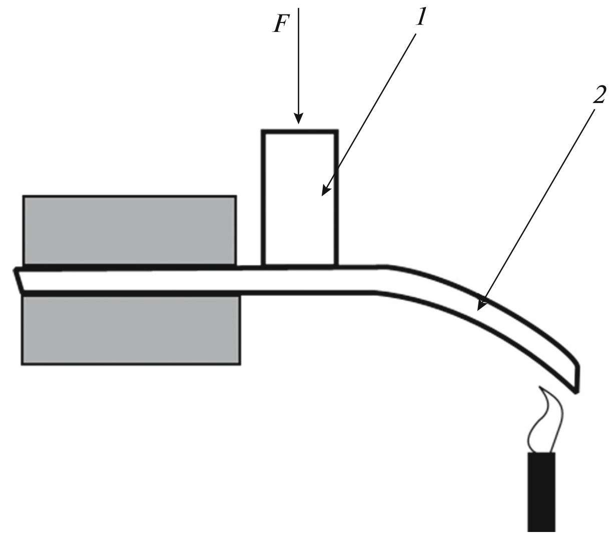

Fig. 7. Diagram of the device used in testing a strained plate: (*1*) AE sensor; (*2*) sample.

remained virtually constant during cooling in the sixth through tenth cycles.

It should be noted that in almost in all our experiments (except for the first thermal cycle), the direct transformation was accompanied by a higher number of signals than the reverse transformation. It appears that the higher Young modulus and lower internal friction of the material in the austenitic state allow us to capture the acoustic signals that accompany transformation in a large volume.

It was shown in [11] that thermal cycling can shift the temperatures of phase transformation, so the thermal cycling of each sample was performed with ten minutes of holding it at −10°C prior to each test. The level of AE in this case fell more slowly from cycle to cycle (Fig. 6), and the highest level was noted during the direct transformation in the first cycle.

As was noted in [7], such findings are largely the result of phase hardening. The increase in the level of AE after keeping samples at room temperature could be due to a change in the phase composition (i.e., an increase in the content of martensite) over time. In addition, stabilization of their structure was shown in [11] to be accompanied by an increase in the elastic properties of a material, which can also favor an increase in the number of recorded pulses. Lowering the cooling temperature naturally increases the content of martensite at the minimum experimental temperature. The above explains the data obtained upon thermal cycling.

AE under Conditions of the Shape Memory Effect after the Active Straining of Martensite

The development of the shape memory effect when we heated the plate altered the AE. The scheme of our experiment is shown in Fig. 7. The distance from the center of the sensor to the edge of the plate was 35 mm. The bend zone was at a distance of 15 mm from the edge of each sample.

Figure 8a shows the pattern of AE development in an unstrained sample. Bending of the plate results in the first group of signals appearing in the range of 50–60 s (Fig. 8b). The recovery of the linear profile of the plate was noted in this time interval; i.e., the shape memory effect developed. The group of signals caused by the transformation in the zone of contact with the sensor appears much later (Fig. 9).

Figure 10 shows the plots of the averaged spectral density of the three main groups of AE signals and their distribution over the energy–median frequency

Table 2

No.	N_{heat}	N_{cool}	N	t_0
First series of thermal cycles				
1	960	377	1337	75
2	62	491	553	120
3	67	460	527	110
4	60	384	444	130
5	51	179	230	125
Repeated series of thermal cycles				
6	1034	140	1174	100
7	32	134	166	130
8	38	133	171	150
9	37	137	174	100
10	30	158	188	105

N_{heat} is the number of AE signals upon heating; N_{cool} is the number of AE signals upon cooling; N is the total number of signals per thermal cycle; and t_0 is the initial moment of AE recording.

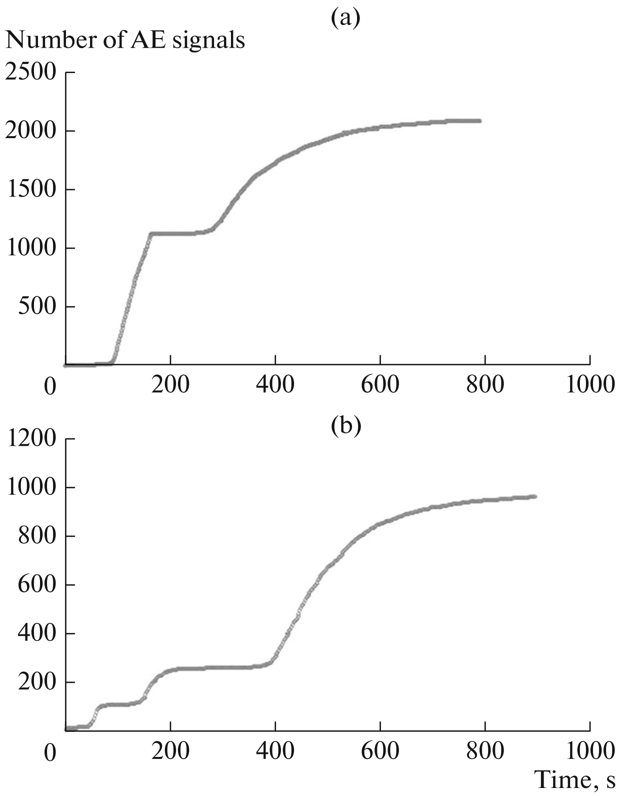

Fig. 8. Total AE count upon the thermal cycling of (a) unstrained and (b) bent plates.

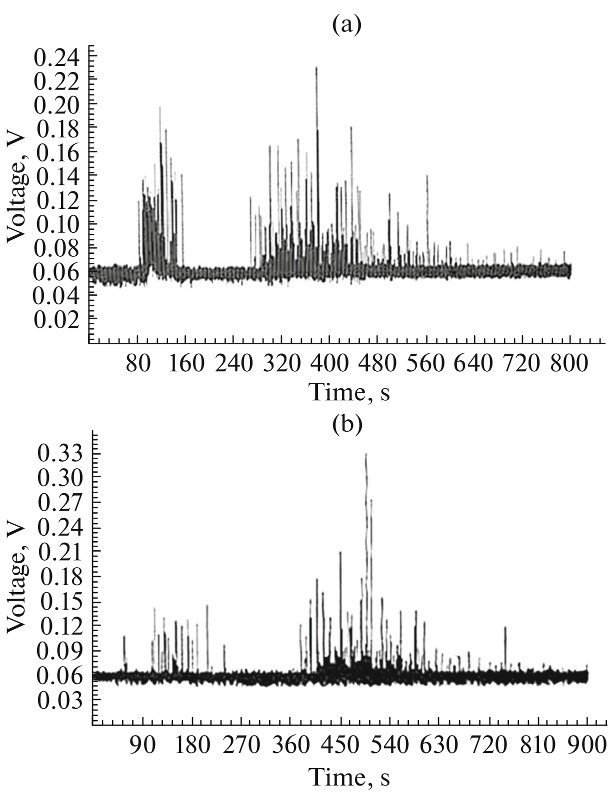

Fig. 9. AE upon the thermal cycling of (a) unstrained and (b) bent plates.

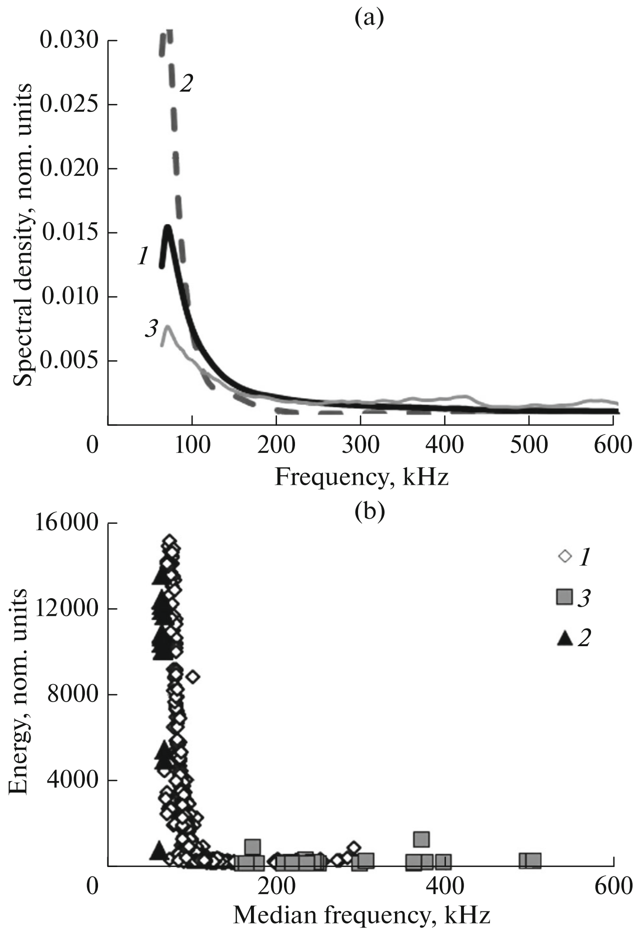

Fig. 10. (a) Spectral patterns of the main types of AE signals upon the thermal cycling of our Ti–Ni alloy sample and (b) the arrangement of these signals in the space of energy–median frequency properties. (*1*, *2*) Main groups of signals upon direct and reverse martensitic transformations and (*3*) signal generated upon the SME.

plane. The signals caused by the shape memory effect (group *3*) differ fundamentally in their spectral characteristics. Their spectral density function not only has a maximum at about 70–80 Hz (as in the two main low-frequency groups of signals) but additional peaks at higher frequencies of 420 and 570 kHz as well. These signals had low energy and median frequencies in the range of 150 to 500 kHz (Fig. 10b).

Results that are interesting from an engineering point of view were also obtained in a series of sequential thermal cycles of our strained samples. In these experiments, the sample was cooled to −10°C after heating the bent plate and recovering the straight profile, and then strained in the previous zone. The plate was then heated again until the shape was recovered once more. The zones of SME development in the cycling tests were shown to generate acoustic signals with intensities that fell from cycle to cycle (Fig. 11).

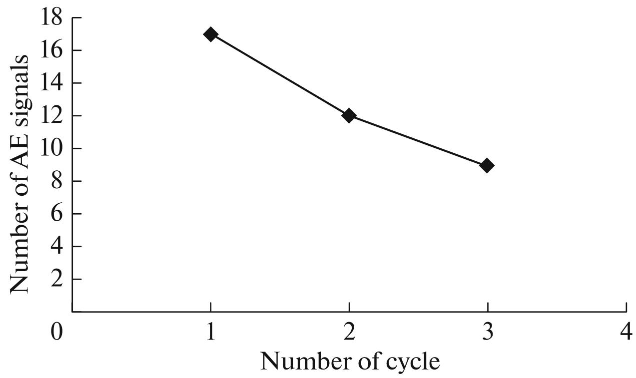

Fig. 11. Change in AE accompanied by the SME in the first three thermal cycles.

Our data show that the AE can be used to identify the processes of lattice rearrangement that accompany those of strain in titanium nickelide.

CONCLUSIONS

Our data show that AE can be used to identify zones of phase transformation in real time. Features of the spectral characteristics of the acoustic signals that accompany the strain processes of the shape memory effect can offer additional opportunities to assess the operational state of the materials of load-bearing members and drives made of shape memory alloys.

AE could therefore be a very sensitive tool in developing procedures for monitoring the status of load-bearing members that operate with the shape memory effect, though this would require further studies of the problem.

ACKNOWLEDGMENTS

This work was supported by the Russian Foundation for Basic Research, project no. 15-01-07657.

REFERENCES

1. V'yunenko, Yu.N., in *Sbornik materialov Mezhdunarodnogo nauchnogo simpoziuma "Perspektivnye materialy i tekhnologii"* (Proc. Int. Sci. Symp. "Advanced Materials and Technologies"), Vitebsk, 2011, p. 182.
2. Kravchenko, Yu.D., Petrov, A.A., Razov, A.I., et al., in *Sbornik materialov XXXV seminara "Aktual'nye problemy prochnosti"* (Proc. XXXV Seminar "Topical Issues of Strength"), Pskov, 1999, p. 260.
3. *Regulatory Document 03-131-97: Guidelines for Acoustic-Emission Inspection of Vessels, Devices, Boilers, and Industrial Pipelines*, 1997.
4. Egle, D.M., Tatro, C.A., and Brown, A.E., *Mater. Eval.*, 1981, vol. 39, no. 11, p. 1037.
5. Muravin, G.B., Simkin, Ya.V., and Merman, A.I., *Defektoskopiya*, 1989, no. 4, p. 9.
6. Merson, D.L., Razuvaev, A.A., and Vinogradov, A.Yu., *Russ. J. Nondestr. Test.*, 2002, vol. 38, no. 7, p. 508.
7. Potekaev, A.I. and Plotnikov, V.A., *Akusticheskaya dissipatsiya energii pri termouprugikh martensitnykh prevrashcheniyakh* (Acoustic Energy Dissipation upon Thermoelastic Martensitic Transformations), Tomsk: Izd. Nauchno-Tekh. Lit., 2004.
8. Plotnikov, V.A., Gryaznov, A.S., and Kharlamov, I.V., *Fundam. Probl. Sovrem. Materialoved.*, 2014, vol. 11, no. 4/2, p. 633.
9. Plotnikov, V.A. and Gryaznov, A.S., in *Materialy XLVII Mezhdunarodnoi konferentsii "Aktual'nye problemy prochnosti"* (Proc. XLVII Conf. "Topical Issues of Strength"), Nizhny Novgorod, 2008, part 1, p. 199.
10. Likhachev, V.A., Kuz'min, S.L., and Kamentseva, Z.P., *Effekt pamyati formy* (Shape Memory Effect), Leningrad: Leningr. Gos. Univ., 1987.
11. Sibirev, A.V., Belyaev, S.P., and Resnina, N.N., in *Sbornik materialov XXII Peterburgskikh chtenii* (Proc. XXII St. Petersburg Readings), St. Petersburg, 2016, p. 345.

Translated by K. Utegenov