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SYSTEMATIC STUDY OF THE MARTENSITIC TRANSFORMATION IN A Cu–Zn–Al ALLOY. REVERSIBILITY VERSUS IRREVERSIBILITY VIA ACOUSTIC EMISSION

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ABSTRACT

The reproducibility and instability of thermal cycling in a restricted temperature domain within the range of the martensitic transformation in a Cu–Zn–Al alloy (68.5 : 14.9 : 16.6 at%) have been studied by means of the acoustic emission (AE) technique. The results indicate reproducibility of the AE bursts produced during the cooling and heating processes and the intrinsic instability related to small thermal perturbations which overlap on cycling.

INTRODUCTION

The acoustic emission (AE) released during martensitic transformations leads to differing results depending on the observation performed (see, e.g., ref. 1 and related references). The observations, including complete transformation cycles [2,3], present an AE with stochastic characteristics and a globally similar pattern. Furthermore, observations on martensite microplates (first plates of martensite appearing in a β-matrix) indicate a good reproducibility in the associated acoustic emission [4]. Small changes in temperature during cycling induce instability in the transformation, i.e., important changes in the way in which plates transform [5].

Some devices are designed for simultaneous observation and measurement [6]. Previous results on optical observation and simultaneous AE detection for martensite microplates show that AE is not directly connected with the transformed mass and that AE bursts are related to discontinuities

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in the growing/shrinkage of the plates i.e, AE is indicative of the dynamics of the transformation [4]. Further observations on martensite plates are clearly in agreement with the connexion between AE and accelerations in the growing/shrinkage of martensite domains [7]. The associated AE signals may have very low amplitudes, being only detectable if the experimental set-up allows signal-to-noise ratios of up to 10 000 [4].

On the other hand, global calorimetric measurements (thermal cycles usually including Mf and Af) show the evolution of the transformation dynamics with the number of cycles. Also, a particular behaviour is observed when working with trained material cut with different surface orientations [8]. A detailed analysis of the calorimetric/acoustic response associated with two consecutive cycles makes an accurate connexion between them difficult [9]. Work carried out with surface martensite (SM) by means of transmission electron microscopy allows the effects that it has on the variants produced during the transformation to be established [10]; i.e., a perturbator effect of its "pure spontaneity" in producing the variants in thin foils of material associated with the orientation of the plate with respect to the axes. Changes in the AE of the microplates and in its dimensions or shape seem to be associated with changes in the domains of the SM, which modifies the interactions with respect to the rest of the lattice [4,5].

This work deals with the stability and reproducibility of the acoustic activity associated with thermal cycling of the material in a temperature range where both phases (martensite and β) coexist. It has been carried out by examining the reproducibility on cycling and the instability in the transformation behaviour after small deviations of the extreme temperature which overlaps on cycling. AE has been used to indicate the dynamic evolution of the material.

EXPERIMENTAL

The experimental system, which has already been described [4,7], allows thermal cycling of the material by means of a Peltier plate. The Peltier effect acts on a cooper plate which serves to homogenize the temperature changes ensuring a homogeneous cooling/heating without significant gradients within the sample. The system allows simultaneous optical observation (microphotograph or videomagnetic tape) [11] and acoustic activity detection via a Canberra 35 multichannel analyser and a Prowler-Norland digital oscilloscope. The acoustic signals are amplified by a Bruel&Kjaer chain (preamplifier and amplifier) and filtered in a band pass from 50 kHz to 2 MHz. A sample of Cu–Al–Zn (68.5 : 14.9 : 16.6 at%) alloy has been used, loaded with a slight external stress to increase Ms to above room temperature, so avoiding condensing water. The applied stress, near the [100] axis, also favours the formation of a single martensite variant.

RESULTS AND DISCUSSION

Previous works analysed the acoustic response of the system for relatively large thermal cycles showing satisfactory reproducibility in its global acoustic emission [7–11].

As shown in Fig. 1, thermal cycling has been repeatedly performed between $T_{min} = 37.02 \pm 0.07\,°C$ and $T_{max} = 41.12 \pm 0.09\,°C$. In this temperature range the growing/shrinkage of a set of microplates can be observed, with an average thickness change of 1 μm and an estimated maximum thickness of 2 μm. Figures 2 and 3 show the micrographs corresponding to the extreme temperatures. The AE associated with the growing/shrinkage of these microplates is simultaneously recorded in a multichannel analyser and in a digital oscilloscope. Two thermal perturbations, $\Delta T = 2.8\,°C$ and $\Delta T' = 5.4\,°C$, were introduced within the thermal cycling, thus delimiting three zones labelled zones 1, 2 and 3 (Fig. 1). The overall amplification was 50 dB in zone 1, either 50 or 60 dB in order to study the amplification effect in zone 2, and 60 dB in zone 3.

Figures 4–7 show the AE ring-down counting obtained in the multichannel analyser (MCS mode) for the three zones indicated in Fig. 1. Figure 4 corresponds to zone 1, with a 50 dB amplification. The AE peaks which appear during each cooling are practically identical. The average number of counts over 21 cooling runs is $N = 66 \pm 4$. Global counting during heating gives an average value over 19 heating runs of $N = 533 \pm 8$. Figures 5 and 6 correspond to zone 2 of cycling, after the first thermal perturbation. Figure 6 shows cycles carried out with different amplifications: 50 dB in the first part and 60 dB in the second. The same peaks are observed for both amplifications, though an increase in the total number of counts, due to more signals crossing the multichannel sensibility threshold, can be observed for the higher amplification. At 60 dB some small signals not observed at 50 dB are detected during cooling. The average counting of the mean cooling peak is now $N(50) = 66 \pm 4$ and $N(60) = 171 \pm 12$. The average values on heating are $N(50) = 556 \pm 30$ and $N(60) = 4242 \pm 165$. These average values are performed over 26 cycles at 50 dB and 15 cycles at 60 dB.

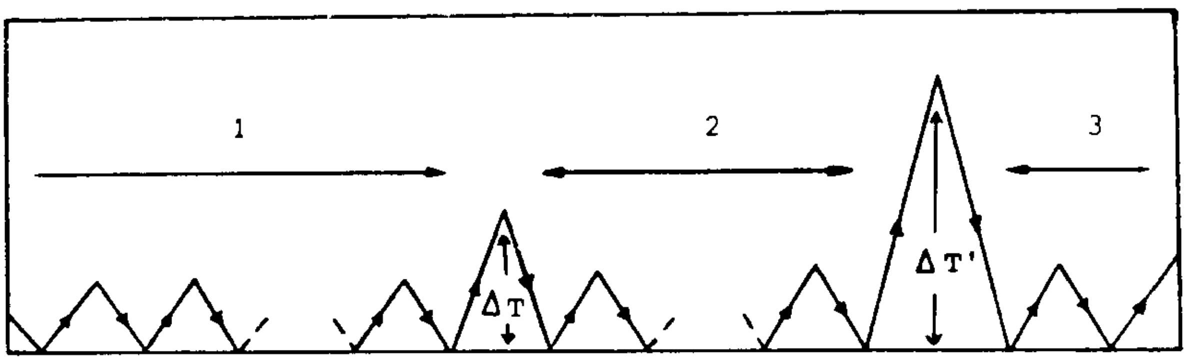

Fig. 1. Thermal cycling.

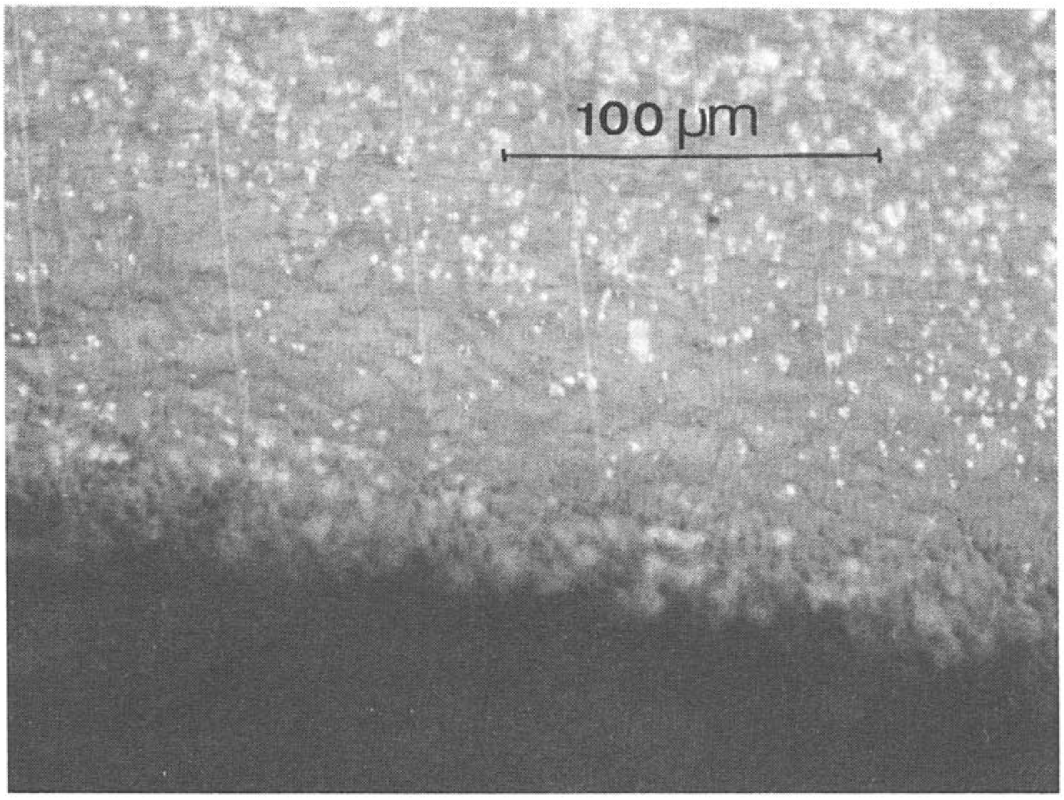

Fig. 2. Photomicrograph corresponding to the minimum cycling temperature.

The effect of a change in amplification was studied by means of digitized bursts for determined AE peaks (Table 1). The analysed pulses on the digital oscilloscope were coherent with the amplification change. From Table 1 the amplification change can be evaluated as 10.7 ± 0.7 dB which is very close to the nominal value of 10 dB. The increase in counting of the Canberra

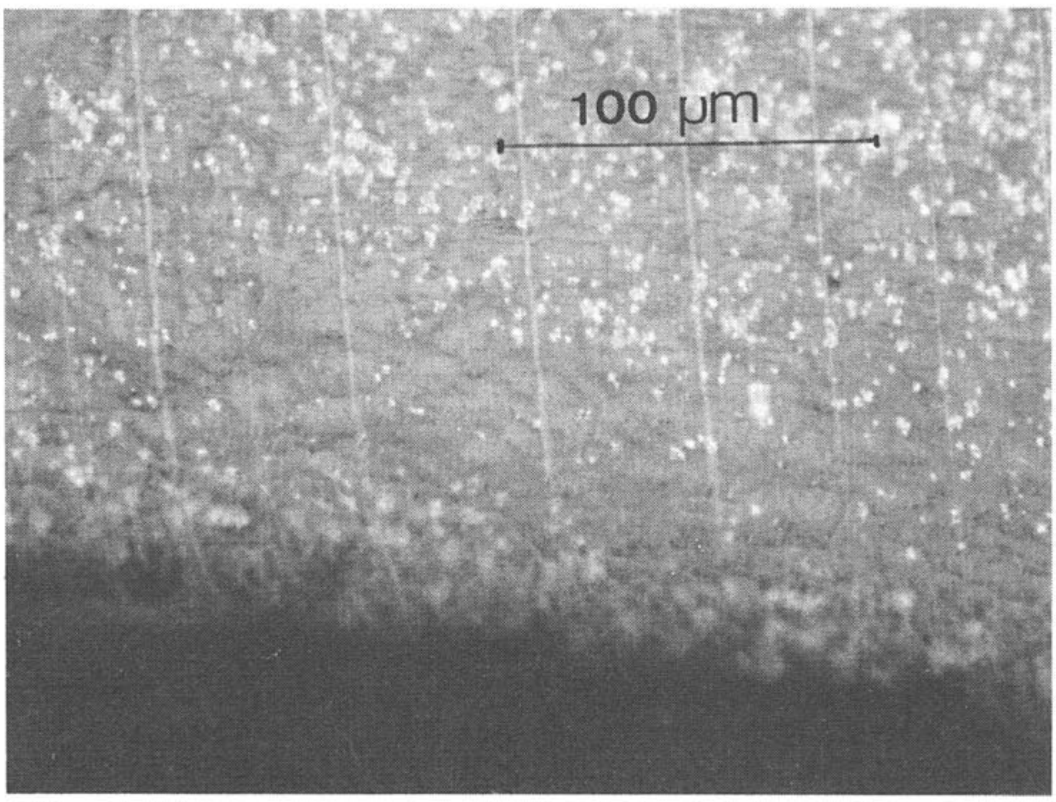

Fig. 3. Photomicrograph corresponding to the higher cycling temperature.

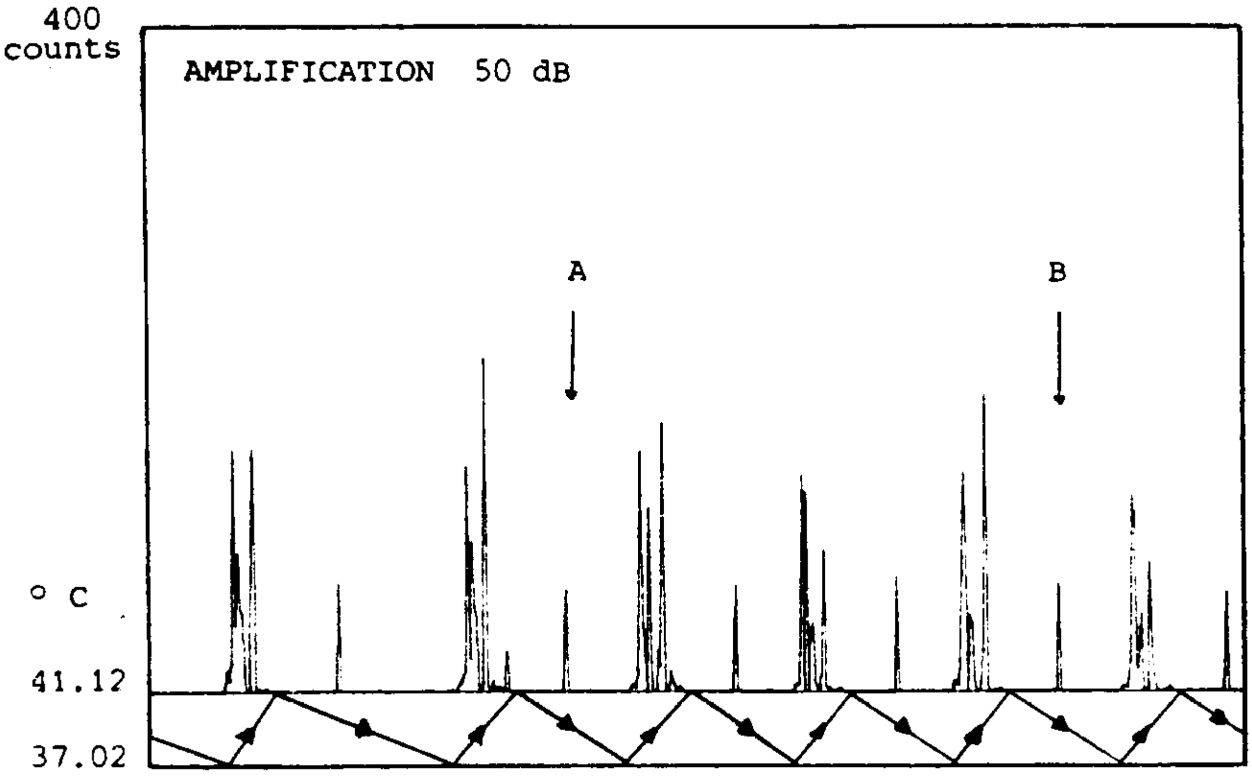

Fig. 4. Acoustic activity vs. temperature in successive cycling corresponding to zone 1.

multichannel analyser using the MCS mode when the amplification increases is not linear since it depends on the amplitude distribution of the acoustic signals.

In Fig. 7, corresponding to zone 3 after the second thermal perturbation, an important change in behaviour can be seen on cooling. In this case the above-mentioned pulse has "broken down" into two different signals which

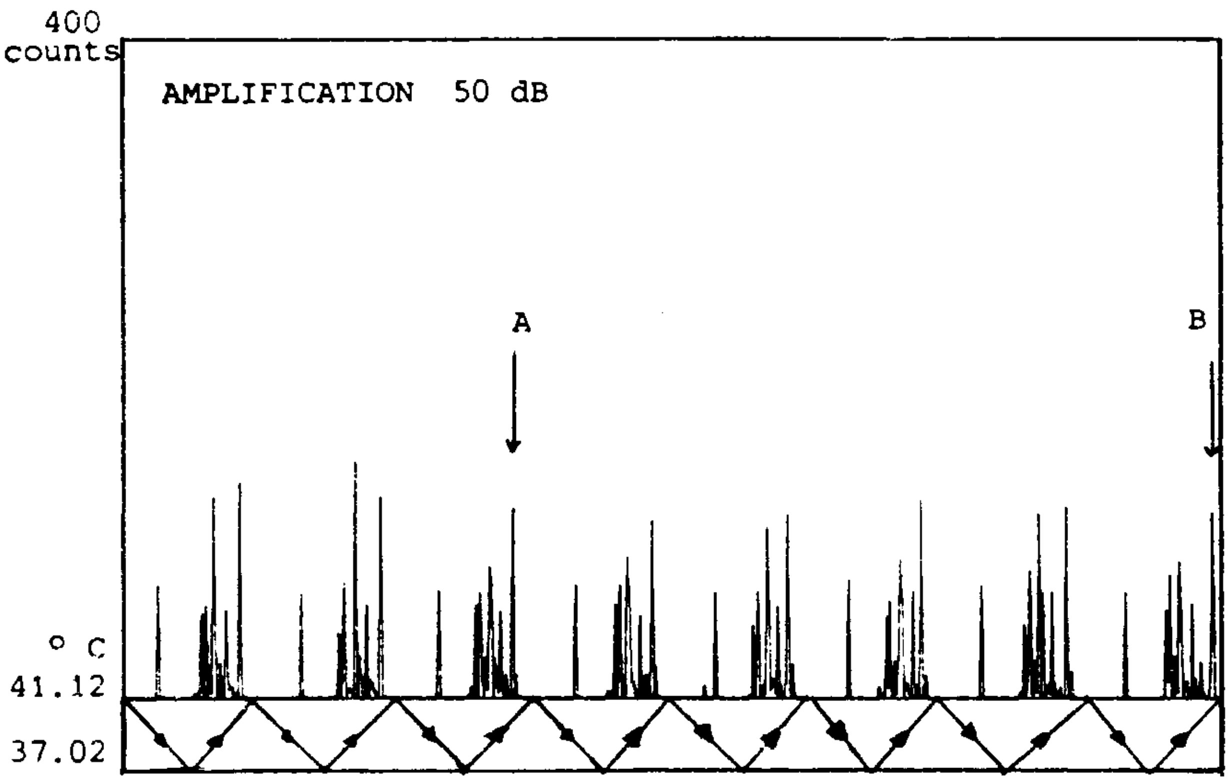

Fig. 5. Acoustic activity vs. temperature in successive cycling corresponding to zone 2.

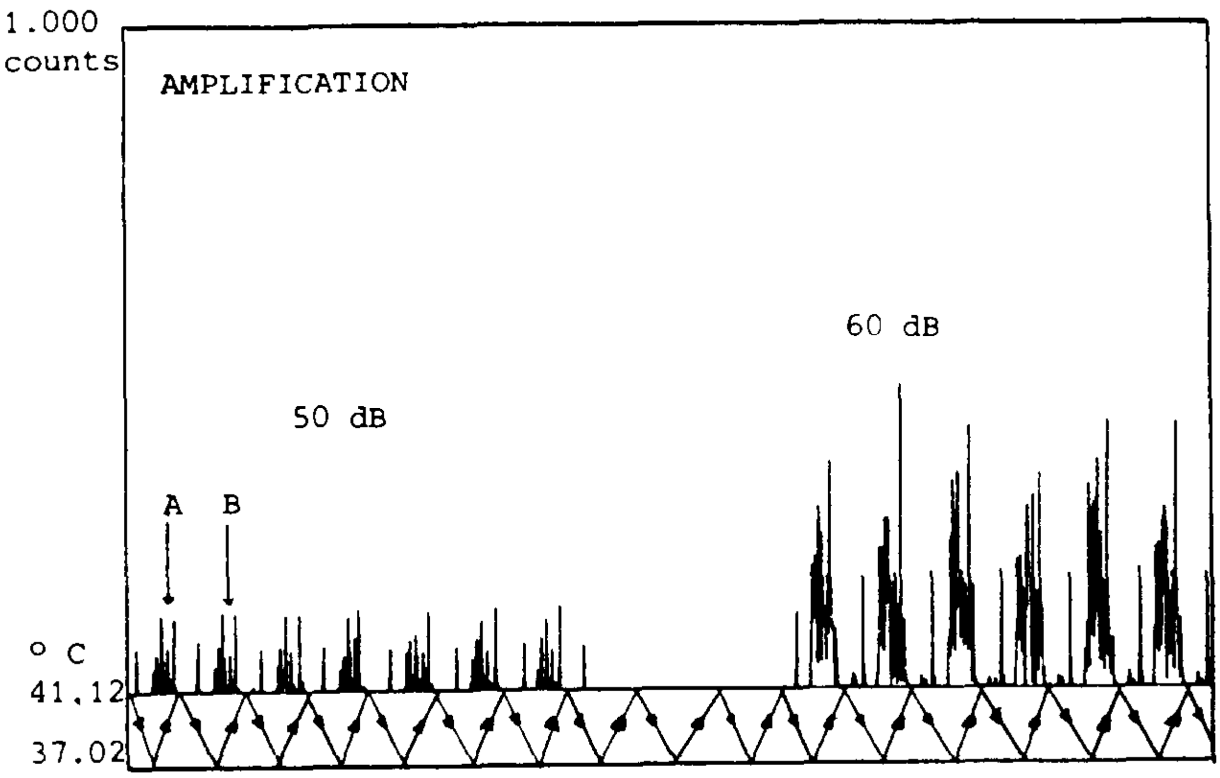

Fig. 6. Acoustic activity vs. temperature in successive cycling corresponding to zone 2 with two different amplifications.

are very close in temperature and which can change its relative position as shown in Fig. 7.

Table 2 presents some results corresponding to the peaks observed on cooling.

Figure 8 presents typical bursts (time response and Fourier transform) corresponding to cooling peaks in zone 1, labelled in Fig. 4. There is total

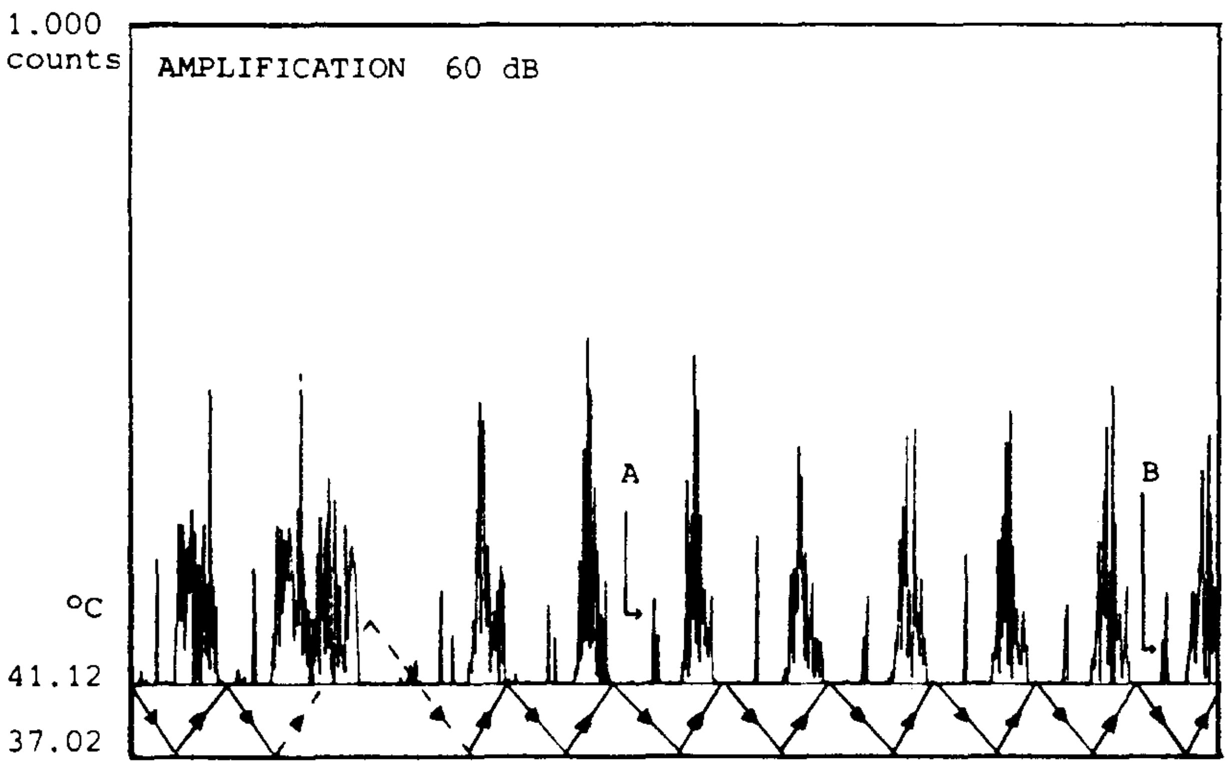

Fig. 7. Acoustic activity vs. temperature in successive cycling corresponding to zone 3.

TABLE 1

Acoustic bursts appearing during cooling in zone 2 [a]

50 dB				60 dB	
k	y (V)	k	y (V)	k	y (V)
F2017	1.13	F2023	1.17	G2011	4.10
F2018	1.17	F2024	1.17	G2012	3.87
F2019	1.17	F2025	1.36	G2013	4.22
F2020	1.13	F2026	1.28	G2014	4.06
F2021	1.17	F2027	1.21	G2015	4.06
F2022	1.13			G2018	4.14
$\bar{y}(50) = 1.19 \pm 0.07$				$\bar{y}(60) = 4.07 \pm 0.11$	

[a] k = identification name of the file, y = maximum amplitude of the burst and $\bar{y}$ = average value with its standard deviation.

agreement between time and frequency for these pulses. Figure 9 presents two kinds of pulses appearing on cooling in zone 3 and labelled in Fig. 7. These pulses are different from those associated with cooling in zone 1, i.e., the thermal change $\Delta T'$ induces a new dynamic transformation. In this case it was not possible to establish an unequivocal relation between the cooling bursts observed in zones 1 and 2 and those of zone 3.

Cooling in zone 2 gives AE peaks which are practically identical to those corresponding to zone 1, although the temperature at which these bursts appear are slightly different in each zone ($T = 38.36 \pm 0.36\,°$C in zone 1 and $T = 37.82 \pm 0.05\,°$C in zone 2). A change in the AE detected on heating is also observed, implying a modification in the transformation. As temperature has been slightly modified, it is possible that the internal stress distribution has changed inside the material without a modification of the pinning which generated the mean AE cooling peak. Heating shows a more complex pattern and selected pulses from Figs. 5 and 6 are shown in Figs. 10 and 11. Figure 10 shows high amplitude bursts labelled in Fig. 5. Figure 11 shows homologous peaks different from the preceding ones, which are labelled in Fig. 6. The homologous bursts are almost coincident, with noticeable differences between non-homologous bursts.

The actual connection between AE and transformation pinning [4] points out the existence of several pinning levels, which can be related to AE intensity. By increasing the amplification, the number of acoustic bursts increases. Detailed observation of the transformation shows that a large number of discontinuities are present, even if the transformation is thermoelastic, as in the present case ($\beta \rightleftarrows \beta'$). Thus, small changes in pinning readily lead to irreversibilities, i.e., deviations from thermoelastic behaviour.

Our observations indicate that AE can easily be modified and that its modification indicates the existence of changes in the transformation mechanisms. Indeed, AE is very sensitive to small changes of temperature and/or

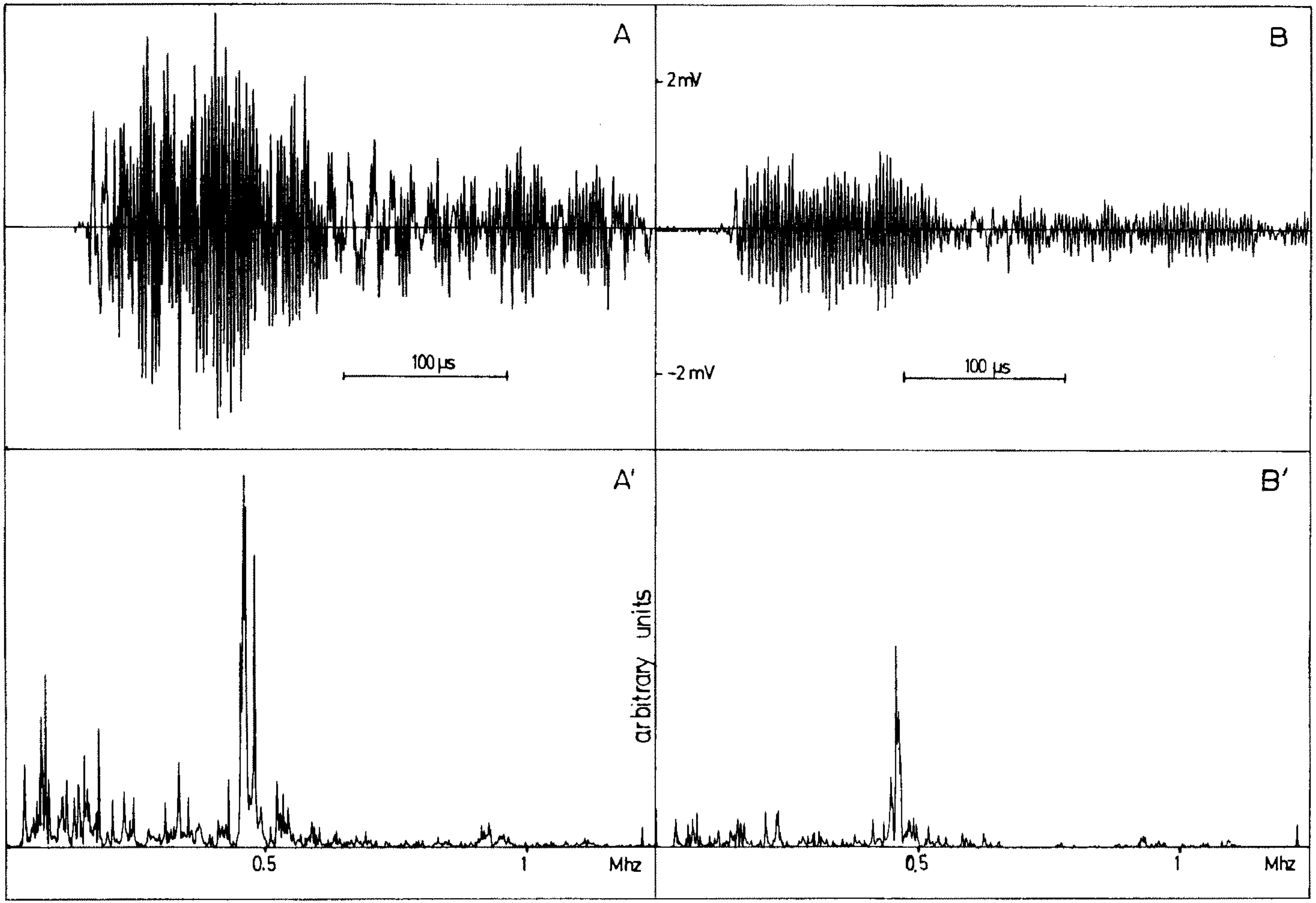

Fig. 8. Time and frequency response of acoustic signals labelled in Fig. 4. Vertical scale values in A and B refer to input units before amplification.

TABLE 2

Acoustic emission peaks appearing on cooling [a]

Zone 1			Zone 2 (50 dB)			Zone 2 (60 dB)			Zone 3		
n	N	T (°C)	n	N	T (°C)	n	N	T (°C)	n	$N1$	$N2$
1	65	38.08	1	65	37.76	1	168	37.82	1	144	74
2	67	38.21	2	73	37.82	2	175	37.83	2	122	72
3	71	38.47	3	63	37.80	3	176	37.80	3	131	76
4	65	38.52	–	–	–	4	171	37.82	4	226	– [b]
–	–	–	5	69	37.80	–	–	–	5	136	72
–	–	–	–	–	–	11	168	37.82	6	198	– [b]
18	73	38.10	23	63	37.82	–	–	–	7	68	122
19	71	38.12	24	65	37.80	23	161	37.82	8	67	139
20	69	38.10	25	69	37.81	24	193	37.85			
21	62	38.10	26	66	37.82	25	178	37.82			

[a] n = cycle number, N = number of counts on the multichannel (MCS mode), T = temperature at which the peak appears, $N1$ = number of counts of the first peak, $N2$ = number of counts of the second peak.

[b] Overlapping of the two peaks on the same channel.

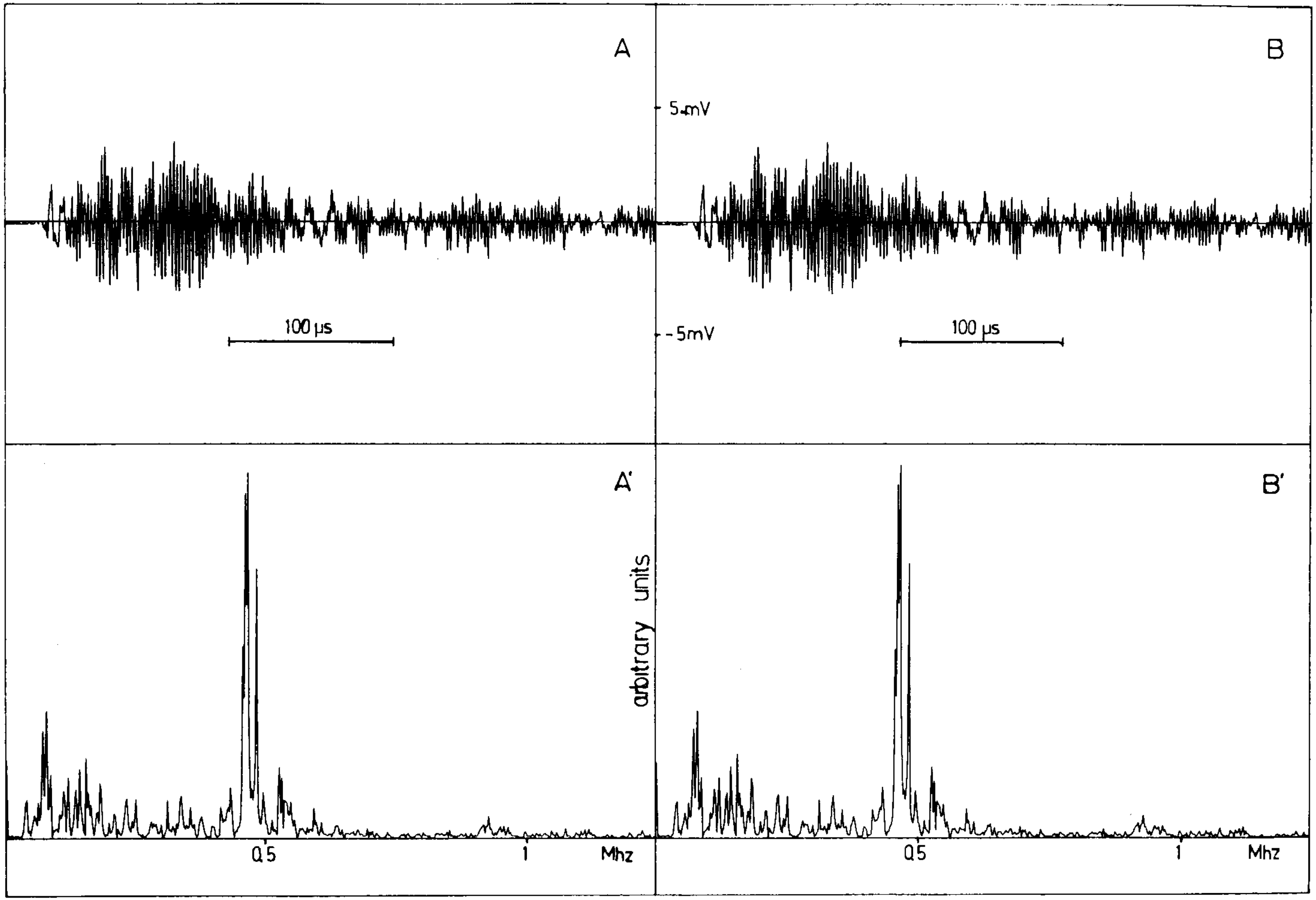

Fig. 9. Time and frequency response of acoustic signals labelled in Fig. 7. Vertical scale values in A and B refer to input units before amplification.

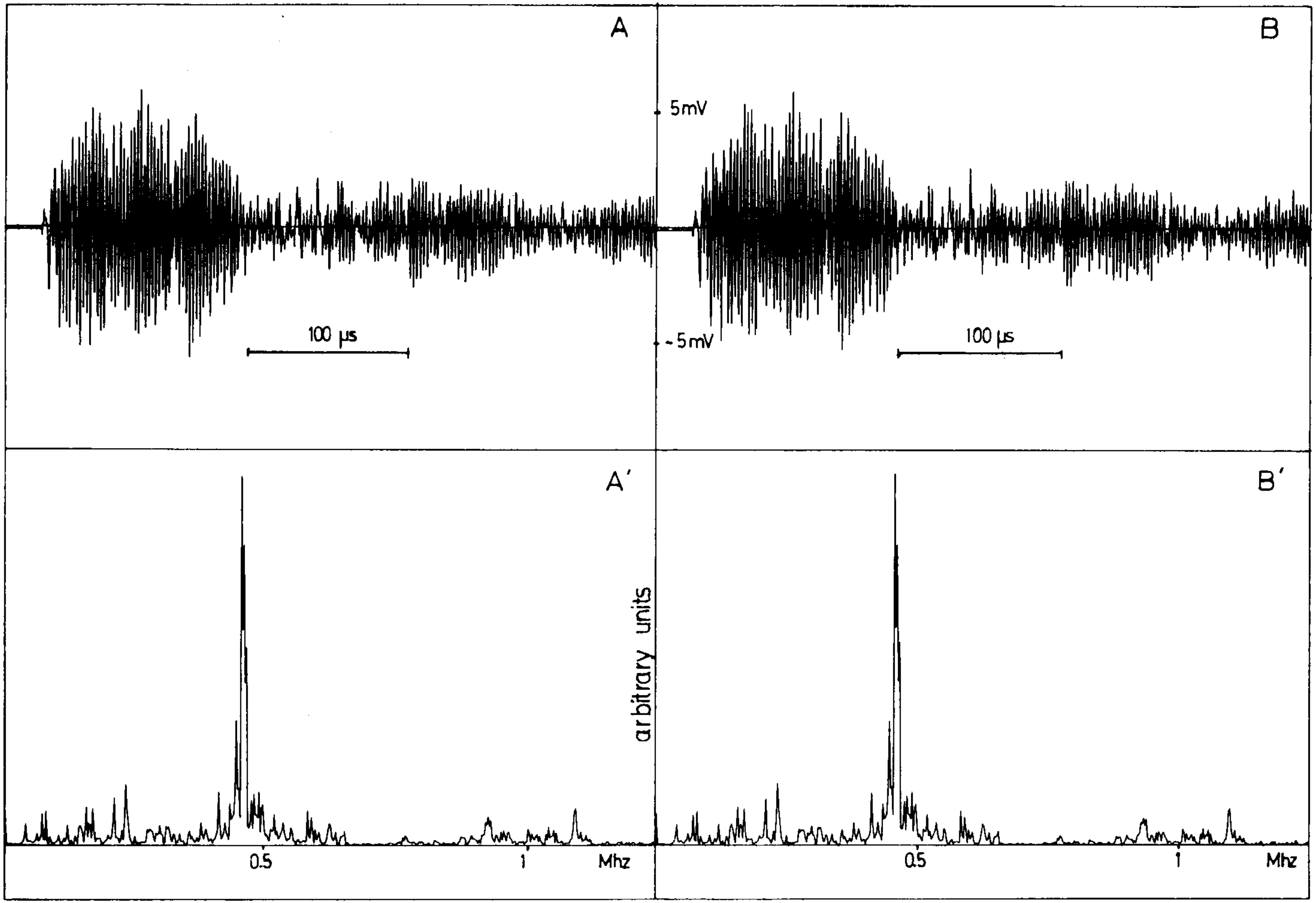

Fig. 10. Time and frequency response of acoustic signals labelled in Fig. 6. Vertical scale values in A and B refer to input units before amplification.

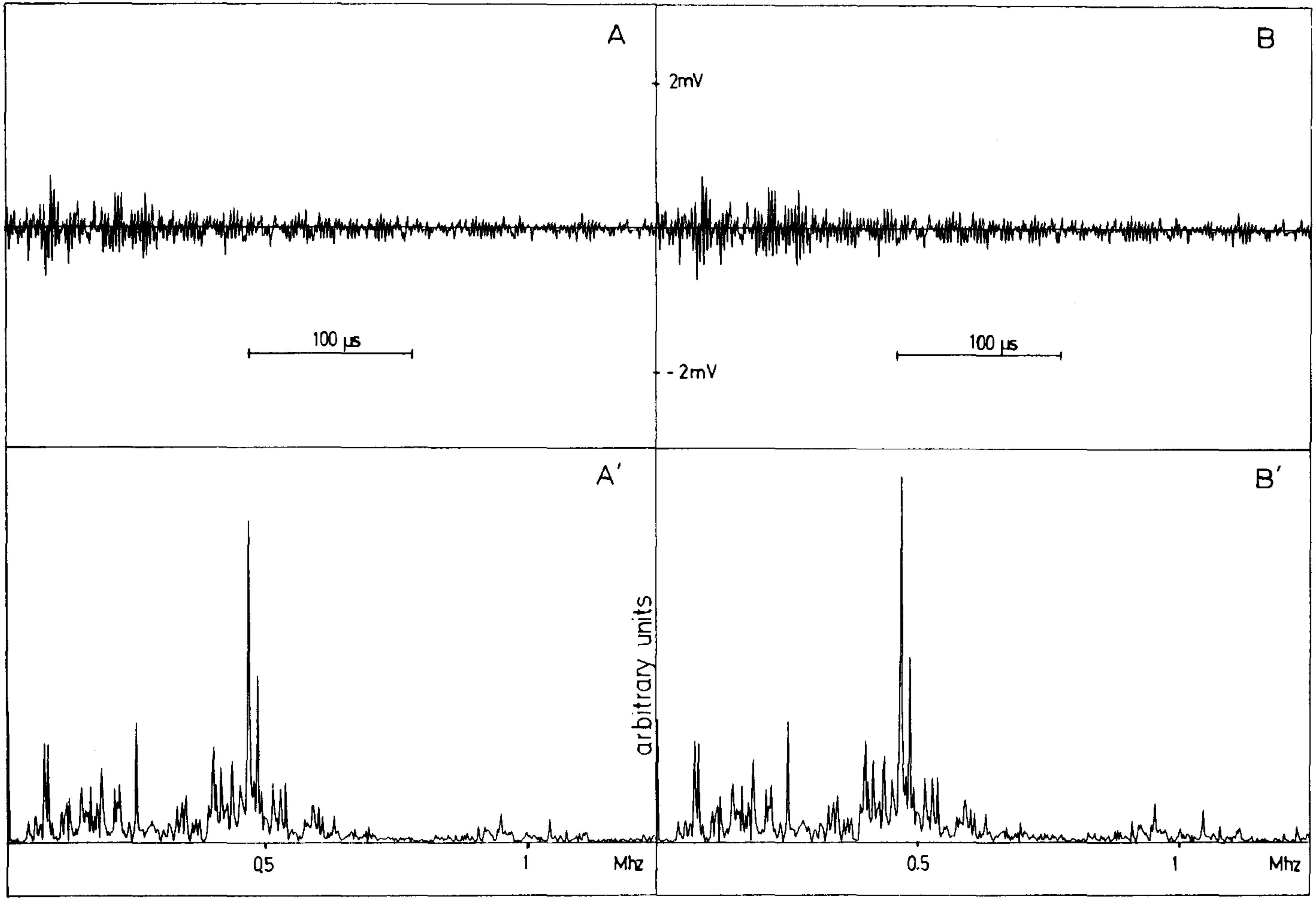

Fig. 11. Time and frequency response of acoustic signals labelled in Fig. 5. Vertical scale values in A and B refer to input units before amplification.

the applied stress (easily changed with the stressing device). Thus, the reversibility of the transformation can be enhanced by eliminating the causes of instability in the AE.

From our experimental observations we establish the following.

(1) The AE bursts are well located in temperature, and its amplitude and characteristic frequencies are reproducible for several cycles in the same domain. Therefore, in terms of the AE, a satisfactory reproducibility of the growing/shrinkage cycles is observed, for an important set of martensite plates.

(2) After small fluctuations of temperature above the cycling domain an instability appears in AE. This implies changes in the transformation not visible in this case by optical microscopy. If the material is submitted to supplementary mechanical and/or thermal stresses, important visible changes in the transformed zones and thus in the associated AE are observed [4,7,11].

These results suggest the following conclusions.

(1) The homogeneous thermal action of the heating/cooling device allows "reversible" transformation/retransformation. Conversely if temperature gradients exist, the transformation can easily change from one cycle to another, beginning in different places, and consequently altering the evolution of the material. An equivalent situation could arise from mechanical actions.

(2) The reversibility of the transformation, in terms of AE, is easily modified by small changes in the temperature range. Surface martensite (SM) acts on the bulk material, but it is irreversibly affected by small changes in temperature. Regarding its own thermal hysteresis and configuration, an interaction between SM and bulk transformation occurs, every change affecting the evolution of the transformation observed in the material. This suggests the convenience of using materials without SM, e.g., surfaces with films.

(3) AE could be associated with the degree of metastability of the domain being transformed. Pinning of the interface generates metastability and induces a difference between the free energies of both phases, probably connected with the intensity of AE. Otherwise, if the transformation proceeds smoothly, the differences between free energies are small and no significant AE is observed. In this case, the results for the first microplates [4] are coherent with those obtained when macroscopic plates of the material transform.

CONCLUSIONS

(1) The growing/shrinkage of martensite plates is reproducible in terms of acoustic emission. Homologous AE bursts for different cycles are equal in terms of time and frequency.

(2) Small changes in cycling temperature induce important changes in the transformation which can be detected by AE.

(3) The reversibility of the process depends on rigorous temperature programming. Fluctuations of $\sim 3°C$ are enough to generate significant changes in AE. In this case, it is necessary that the mechanical stresses applied are reproducible. This suggests that supression of surface martensite, which is very sensitive to temperature fluctuations, could reduce the instability of the material.

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