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Experiments on acoustic emission generated during rapid heating of tin single crystals

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The power of a new acoustic emission technique is explored. It is found that it can be profitably applied to reveal structural transitions and to measure their characteristic temperatures. Information concerning the effect of cold work on structural transitions and on the internal strain state can also be obtained.

1. Introduction

In the last few years the detection of elastic waves emitted, along with dynamical processes developing within a solid (acoustic emission), has become increasingly important in the field of nondestructive testing. This is in spite of the fact that most of the information carried by an acoustic emission signal is very difficult to extract, because a signal is processed by the sample-transducer system before it is detected.

In fact with acoustic emission techniques, phenomena can be investigated which take place on an ultra-fine scale, out of the reach of most experimental methods (dimensional changes a low as 10^{-12} in. can be easily revealed [1]. The detection apparatus is relatively simple, and data are collected in real time. As a result, the information obtainable by simply counting the emitted signals is often very valuable for materials evaluation.

In most acoustic emission experiments, acoustic emission is excited by either of two methods: (1) direct mechanical deformation; (2) temperature change. The latter method has mainly been used to study phase transformations of a martensitic kind [2–4]. Such experiments are performed with heating (or cooling) rates of $\sim 1^\circ C\,min^{-1}$ or less. These rates permit the transformation process to start and to be maintained; at the same time they are low enough so that a study of the transformation versus temperature is not meaningless.

It has been shown in a recent paper [5] that temperature changes can be used to generate acoustic emission in a different way, i.e. by heating with rates as high as $1^\circ C\,sec^{-1}$. It has been found that such a method produces detectable acoustic emission acitvity in tin and zinc single crystals. These materials do not undergo martensitic phase transformations. In any case, no acoustic emission is generated by heating (or cooling) them with the same rates employed for materials that do show martensitic transformations.

It should be noted that acoustic emission has also been detected during welding and its cooling period [6]. So far, however, no attempt has been made, except by Papa *et al.* [5], to use acoustic emission produced by fast temperature changes as an independent method for materials investigation. The aim of the present work is to point out some applications of this experimental method, which presents itself as very simple and versatile. The experiments are carried out on Sn, which showed emission features more suitable for our purpose [5]. First, results concerning the measurement of characteristic temperatures of the acoustic emission generating process are reported, then the response to an impulsive load is examined.

2. Experimental details

Sn samples about 25 mm diameter and 40 mm long, were cut by spark erosion from the same block of

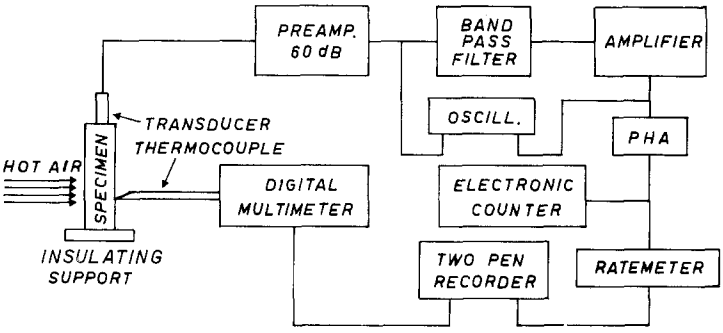

Figure 1 Block diagram of the experimental set-up.

Sn single crystal (99.99% pure, with Zn as main impurity). Fig. 1 shows a diagram of the experimental set up. The specimens were heated to 130° C with a low-speed, hot air flux. The temperature changes were monitored with a copper–constantan thermocouple placed in a small hole drilled in the specimen side; $0.7^\circ\,\mathrm{C\,sec^{-1}}$ was a typical heating rate. The heated samples were cooled with a room temperature air flux; in order to reach lower temperatures a cell cooled with ice was employed.

The acoustic emission signals were picked up with a PZT-5 transducer. It was electrostatically shielded by an aluminium case and electrically isolated from the sample surface by a thin layer of epoxy resin. The latter was contacted to the tin by an acoustic coupling medium (silicone grease).

The transducer output was sent to a 60 dB low-noise preamplifier, followed by a selectable band-pass filter and a variable-gain post-amplifier. The amplifier and preamplifier outputs were monitored with an oscilloscope. Fig. 2 shows the oscillogram of a typical acoustic emission burst. It was found that the major frequency carried by a burst at the preamplifier output was about 200 kHz. However, the best signal-to-noise ratio was obtained with a 40 to 120 kHz band-pass, because of the peculiar frequency content of the external noise.

The counting apparatus was completed with a single-channel pulse height analyser (PHA), an electronic counter, and a ratemeter. The analogue output of the latter, together with the thermocouple signal, was displayed versus time with a two pen recorder. In the present experiments, the PHA was used to provide an adjustable voltage threshold for the ratemeter and the counter.

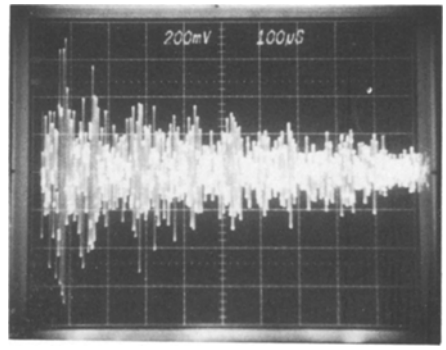

Figure 2 Oscilloscope trace of an acoustic emission burst (preamplifier output).

The ring-down counting technique (i.e. the counting of the number of times a threshold voltage is exceeded by the oscillating transduced output caused by acoustic emission [7]) was usually employed. In one case, however (see Section 3.1), the counting of acoustic emission bursts (or events) was required to obtain a sufficient accuracy.

Fig. 3 schematically shows the experimental procedure. Heating starts from a temperature T_m. When a temperature T_f (between 60 and 80° C) is reached, the emission activity stops, while the heating is kept on up to 130° C. At this point, the counter is reset, and the specimen is cooled back to T_m. In the same figure, T_s and T'_s represent characteristic temperatures, whereas T' is an intermediate temperature (see Section 3.1); N_T is the total count over a single heating run, and N' is the partial count for $T = T'$.*

Specimens were subjected to several hundred identical thermal cycles without any change of the count's mean value and fluctuation having been observed. The total time required by one thermal cycle was typically 5 min.

* The estimated characteristic heat diffusion time across a specimen is about 15 sec, which should produce, during heating, a maximum temperature difference of about 10° C between surface and interior. In particular, this should constitute the major contribution to the observed difference between the temperatures T_f and T'_s (see after).

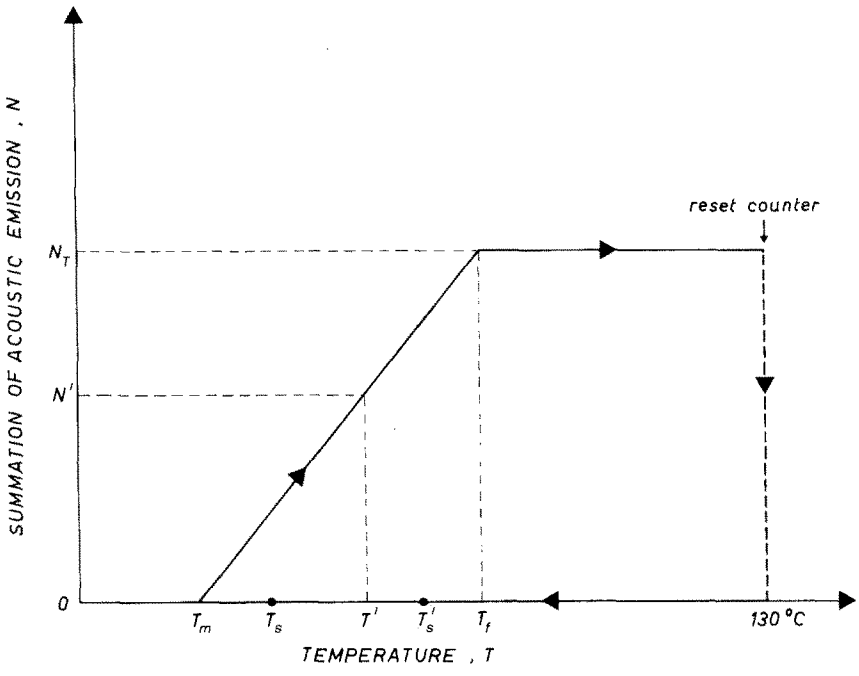

Figure 3 Schema of the experimental procedure.

3. Experimental results

3.1. Characteristic temperatures

Preliminary experiments have shown that acoustic emission activity is negligible if T_m is higher than a characteristic temperature T_s (see Fig. 3). To measure the value of T_s, the total number of events, or acoustic emission bursts, in a single heating run (as given by observations with the oscilloscope) was measured versus the starting temperature T_m for a number of otherwise identical thermal cycles. The results are shown in Fig. 4. A rather sharp transition range (13.7 to 14.9° C) is

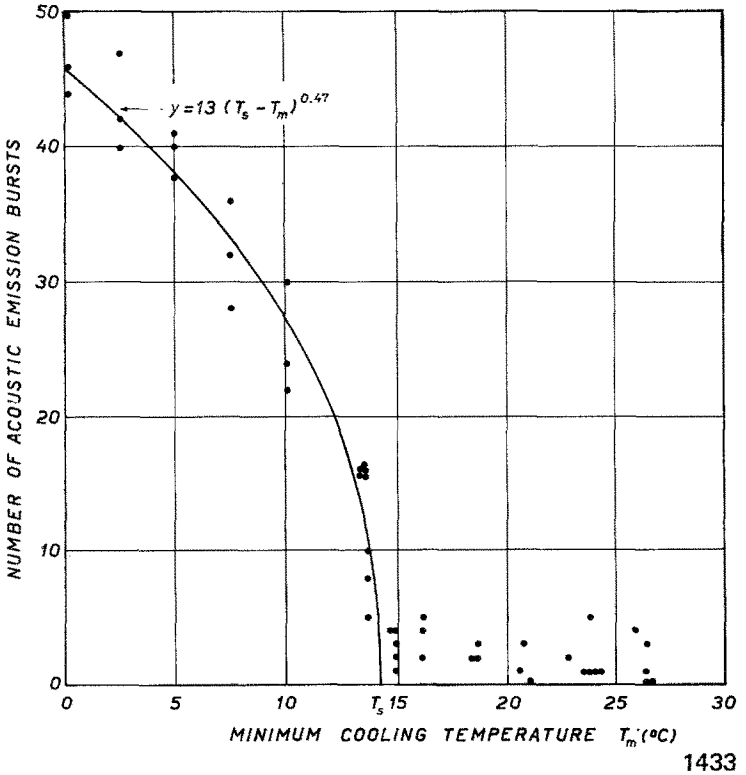

Figure 4 Total number of acoustic emission bursts detected over a single heating run versus minimum cooling temperature. The solid line fits the experimental points for $T_m < T_s$.

observable, so that for lower temperatures a curve rapidly increasing with decreasing T_m fits the experimental points (solid line in Fig. 4); instead, for higher temperatures we observe a sort of *background noise* configuration. This gives the following value for T_s'.

$$T_s = (14.3 \pm 0.6)^\circ \text{C}.$$

This result does not depend upon the time spent at T_m, and it agrees very well with the value of the $\beta \rightarrow \alpha$ transformation point as deduced from rate data [8], measurements with a grey tin–white tin equilibrium electrolytic cell [9], and dilatometry techniques [10].

Experiments have been made in which during a thermal cycle, starting from $T_m = 0^\circ$ C, the heating is stopped for a time interval Δt at a temperature T' larger than T_s (see Fig. 3); then the cycle is completed without any other interruption. In Fig. 5 the ratio $r = (N_T - N')/N'$ of the ring-down counts obtained on heating *above* and *below* T', is plotted versus T' for some values of Δt. The lowest value of Δt (1 min) was chosen in order to be sure

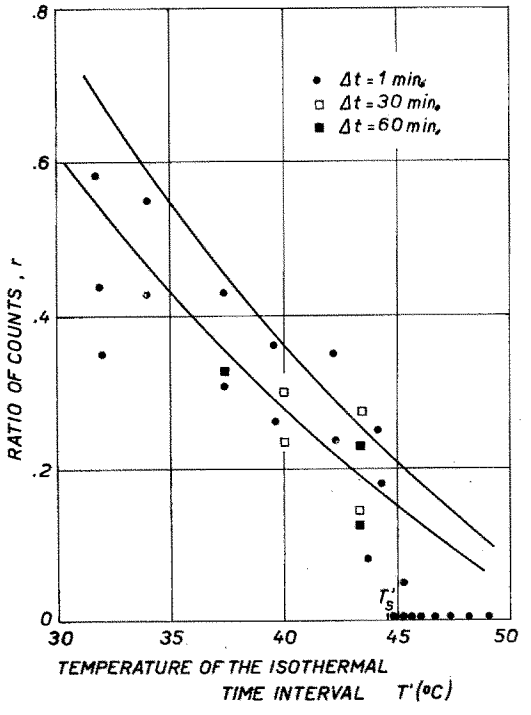

Figure 5 Ratio $r = (N_T - N')/N'$ of the ring-down counts measured after and before an isothermal time interval Δt, versus the temperature T' of the interval, for some value of Δt. Solid lines limit the area containing all experimental points (not shown) for $\Delta t = 0$. For $T' > 45^\circ$ C each plotted point represent several experimental points.

of reaching thermal equilibrium after the heating was stopped. The two solid lines in Fig. 5 limit the area containing the points (not shown in the graph) measured for a great number of uninterrupted ($\Delta t = 0$) heating runs.

The experimental points are too scattered to single out any definite relation among r, T' and Δt. However, a clear discontinuity occurs for temperatures between 44.2 and 44.7° C, so that for higher temperatures, and for $\Delta t \geqslant 1$ min, the emission activity can always be considered absent (for $T' > 45^\circ$ C each point near $r = 0$ in the graph represents several experimental points). This defines a second characteristic temperature T_s'. Because of its value, it may be related in some way to the $\alpha \rightarrow \beta$ transformation [11].

3.2. The effect of an impulsive load

The measured value of T_s makes it conceivable that the observed acoustic emission is closely related to the occurrence of $\alpha \leftrightarrow \beta$ transformation events. It is well known that such a phase change is noticeably accelerated by cold work [12]. It should then be expected that plastic deformation of our Sn samples will influence the acoustic emission response. A systematic investigation on this topic is at present in progress. We limit ourselves to report here the results of a simple experiment which yield some general information on the subject, and also show another interesting effect.

In this experiment several emissions were recorded from an as-cut specimen; it was then cooled to 0° C and dropped from the height of 1 m on to a brass plate. The results are shown in Fig. 6. Curve A represents a typical summation of acoustic versus time during a heating run before deformation. Curve B refers to the heating performed immediately after deformation. Curves C and D were recorded during the second and tenth heating after deformation, respectively. Subsequent heatings produce curves that show only slight deviations from curve D. Two distinct effects are observed, i.e.: (1) the increase of the activity level as a permanent characteristic of the deformed specimen (common part of the B, C, D curves); (2) the appearance of a *background* activity during the first heating after deformation (curve B); it extends well beyond the limits observed for usual emissions (60 to 80° C), and does not repeat itself in the subsequent heatings.

Metallographic observations with an optical microscope showed that the zone directly affected

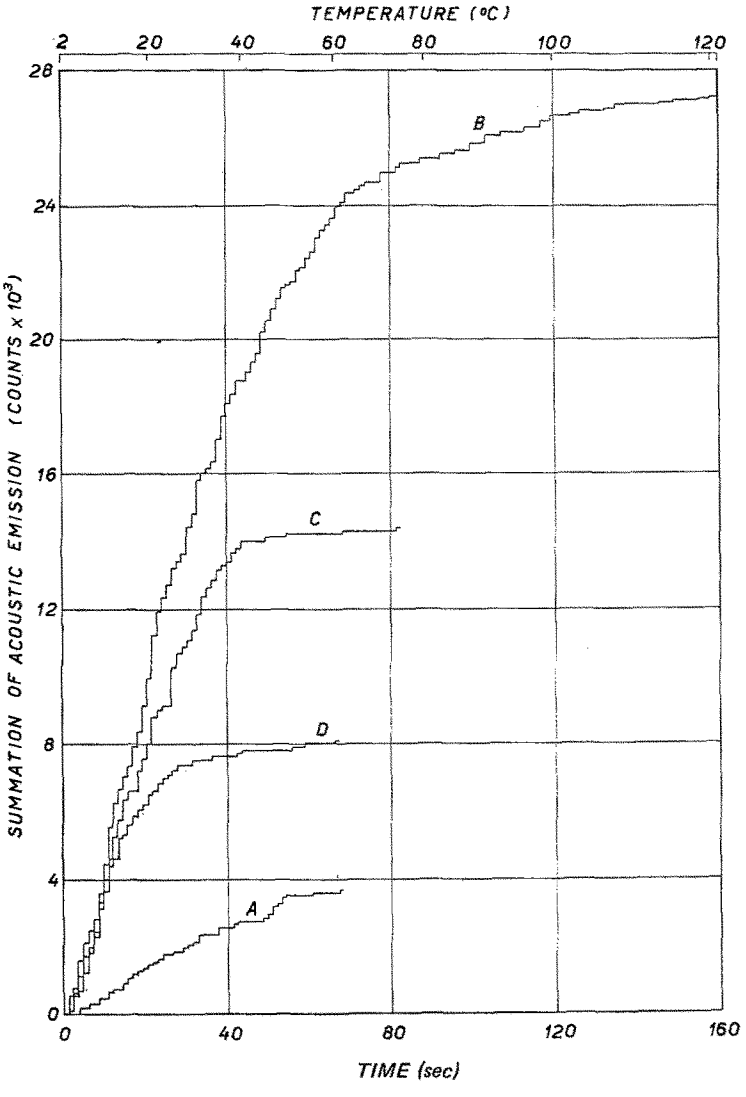

Figure 6 Acoustic emission summation versus time during a heating run, showing the effect of an impulsive load. (A) Undeformed specimen, (B) first heating after deformation, (C) second heating, (D) tenth heating. The instantaneous temperature is approximately shown on the top axis.

by the impact was turned into a polycrystalline region, with grains of about 1 mm in linear dimension. Moreover, some new twin bands depart from this region.

4. Discussion

From the general features of the phenomenon it is deduced that the acoustic emission observed in Sn is essentially associated with a reversible, temperature-induced structural transition. As already mentioned, it appears quite natural to identify such a transition with the $\alpha \leftrightarrow \beta$ phase change. While cooling from T_s to T_m, α-nuclei should form inside the β-Sn sample. During the subsequent rapid heating these nuclei will disappear through a shear-like process induced by thermal stresses producing acoustic emission pulses. In this respect, T_s is the point of the $\beta \rightarrow \alpha$ transformation, whereas T_s' represents the maximum temperature at which we find an α-centre in equilibrium (stable or metastable) with the matrix.

Although this interpretation is supported by the measured values of T_s and T_s' (see Section 3.1), there are two objections: (1) no direct evidence (i.e. with microscopy techniques) has been obtained of the occurrence of the $\beta \rightarrow \alpha$ transformation in our experimental conditions; (2) it is well known that the presence of the α-phase in a β-Sn specimen is detectable only after an undercooling period of hours or days or even years [12]; yet we have observed acoustic emission activity by heating just after a few seconds in slight undercooling conditions.

These facts, however, do not contradict our hypothesis. Actually, because of its high sensitivity, acoustic emission can reveal a single submicroscopic

transformation event. Usual techniques, on the other hand, only interact with either a microscopically observable nucleus, or a sufficiently large number of submicroscopic nuclei so as to produce a detectable total volume increase (dilatometric techniques).

Thus, as a consequence of this work, a reasonable description of the first stages of the $\beta \rightarrow \alpha$ Sn transformation can be qualitatively proposed as follows. In undercooling conditions, submicroscopic α-centres soon nucleate. These nuclei are produced only by the transformation of highly strained regions, so that no increase in the Gibbs energy is introduced by a nucleation event (the $\beta \rightarrow \alpha$ transformation involves a 27% volume increase). No growth takes place, since it produces a large strain energy increase (e.g. the strain energy of an elastic inclusion is proportional to its volume [13]). Finally, the nucleation sites are exhausted after a short time. This is confirmed by the results of experiments on the dependence of the acoustic emission acitivty upon the time spent at T_m ($< T_s$), which showed that the total count, N_T, saturates exponentially with a time constant of about 4 min.

As a result, the transformation will eventually be detected on a microscopic scale only because of the growth of some nuclei on or near the sample surface, just as actually observed [12]. In fact, for these nuclei the strain energy is lower, and a slow process of strain energy release by plastic deformation is more probable to occur.

To complete our comments on the results of Section 3.1, let us note that alternative explanations for T_s' may be proposed. For example, a fraction of the observed acoustic emission could be produced by the motion of dislocations associated with the Zn impurity atoms; hence, T_s' should represent the temperature at which these atoms no longer strain the Sn lattice. However, the fraction of acoustic emission that could not be attributed to the $\alpha \leftrightarrow \beta$ transformation (see Fig. 4 for $T_m > T_s$) seems too small to account for the emission observed from T_s to T_s'; moreover, it may simply be related to the internal strains set up by thermal cycling (see below).

The permanent increase of the acoustic emission level found after plastic deformation (see Section 3.2) is now readily interpreted as due to the increase, produced by the deformation, of the number of strained regions which act as sites for the α-phase nucleation. Thus the experiment shows that the influence of cold work on a structural transition could be profitably investigated with the present technique.

The same experiment points out another possibility of practical application. In fact, the transient component of acoustic emission after plastic deformation (see curve B of Fig. 6) shows that rapid heating can also produce acoustic emission signals associated with processes other than a phase transition. When a ductile crystal undergoes an impulsive load, we observe that some of the generated dislocations arrange themselves in "stable" configurations, such as grain boundaries (see Section 3.2). On the other hand, we expect that a noticeable part of the produced defect configurations remain unstable; under external excitation, rearrangement occurs and strain energy is released. Our experiment proves that this excitation can simply be provided by a sufficiently rapid heating, and that the strain energy release can be detected as acoustic emission. Let us consider as an example a twin nucleus of critical dimensions: when its growth starts, an acoustic emission pulse is produced [14]. It is very likely that similar processes also give rise to the emission observed during heating of Zn single crystals [5], as well as to the signals observed in Sn for $T_m > T_s$ (see Fig. 4). In such cases, indeed, internal deformation caused by thermal cycling should be present.

5. Conclusions

(1) The experimental technique proposed in this paper permits us to reveal a structural transition in Sn single crystals. So far such a transition has not been detected under the same conditions by other methods. Its characteristic temperatures are measured with a reasonable accuracy, and its dependence on plastic deformation can be investigated.

(2) A phase-transition-independent acoustic emission is obtained by rapidly heating an as-deformed specimen. This may lead to a simpe method for establishing the effect of a given macroscopic deformation process on the internal strain state of a material.

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