Acoustic Emission Study of Twinning in Indium Crystals and Lead-Tin Alloys

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Mr. Stephen Van Doren, Dr. Robert E. Green, Jr., Dr. Jan and myself are very fortunate to have the Air Force OSR sponsor this work having to do with acoustic emission. If any of the other three gentlemen were here, he would do an excellent job of describing the circuitry, equipment and techniques, but I am principally interested in revealing to you some unique data which we have developed in these investigations.

Dr. Evans' lecture has been an excellent introduction to my presentation. I would like to say, however, that at one time we were asked if we were for or against evaluation by acoustic emission, and we said that we were against it. The reason we were against it was because of a doubt that some of the test procedures were actually telling us anything about cracking. We, therefore, accepted as a chore to try to do what Dr. Evans said should be done, that is, see if we can discriminate between slip, twinning, fracture in acoustic emission signals.

It was decided to first investigate the emission produced by twinning. Since acoustic emission from twinning is audible, it was presumed that a great deal of information could be gathered with a transducer. Another piece of information caused indium to be selected as the material in which to study twinning. When an indium single crystal of the proper orientation is caused to twin in tension, it can be caused to reverse in compression. That is, you can put a twin in it one way and pull it out the other way. The specimen we used in this study was square in cross section with a 1/8" x 1/8" x 1" gauge length. This small specimen can be twinned and detwinned by loading it with one's fingers. If we cause the twin to develop and we do not continue to stress it so slip
occurs, then we can continue to reverse it back and forth. We have done it as many as 52 times in a specimen with each operation producing an audible click.

What I would really like to do today is just to show you some of the data we have gotten and go through it rather rapidly. Some of the things I have to show you support very well what Dr. Evans had to say.

Figure 1 is a print from a 16 millimeter movie track showing the development of a twin in an indium single crystal. These pictures were taken at about 250 frames a second. While this specimen is being loaded it is still attached to its hot top. There actually will be an array of 4 to 16 crystals all in the same orientation on one hot top. The transducer is put on the hot top, so as we deform these crystals back and forth to cause them to twin and untwin, or move from specimen to specimen, we are sure we never change the coupling of the transducer. The acoustic signal is recorded on a video tape recorder as the specimen is being twinned (or detwinned) and as the twin's appearance and growth is being documented through the motion picture record. Since the movie record and the video tape record can be synchronized in time, it is possible to examine the sonic signature relative to the surface manifestation of the twinning deformation.

If we look at the acoustic impression we get, from a twin that is in tension relative to one in compression, we see a polaric change. The transducer seems to pick up a different effect from a tensile wave than from a compressive wave. This is illustrated in Fig. 2 wherein the first oscillogram shows the pulse produced by a twin formed when the crystal is loaded in compression. The second oscillogram shows the pulse produced when the same volume of indium detwinds by loading the specimen in tension. We have calculated and measured the speed of sound in the indium and run calculations based on the specimen length to see if the observation is the result of a reflective signal, and it is not.

Next, I would like to move to the load displacement curves that were done on a series of alloys that go from a hundred per cent lead to
Figure 1. Consecutive frame prints from 16 mm motion picture track showing development and growth of a spine in single crystal indium.
Figure 2. Oscillographs showing the polarity difference in the acoustic emission signatures made by twins formed in tension and compression. 0.2 volt/cm in amplitude, 0.5 μsec/cm in time.
a hundred per cent tin at 10 per cent intervals across the phase diagram. The specimens tested in this series were square in cross section (1/2" x 1/2" x 3" gauge length) and had button heads at either end which fit special grips in the Instron machine. As each specimen was extended in tension, the acoustic pulses emitted which could be discriminated over the background noise were counted as a function of time. The crosshead speed in these tests is about 0.005 centimeters per minute, and the acoustic emission plotted is in counts per 30 seconds.

Figure 3 shows the record for pure lead. The serrated load-displacement curve should be noted. The lead used in this test is 99.99 per cent pure lead. It should not develop twins on deformation, but we certainly get something that looks like twinning emission.

Figure 4 shows the record for 10 per cent tin which is a solid solution alloy and we are picking up an emission that isn't too different from the one we saw for lead.

In Figure 5, at 20 per cent tin, we notice that we get an early, high count rate and then just some low noise. The load-displacement characteristic in the material is, of course, changing.

Figure 6, illustrating the 30 per cent tin alloy behavior, is not very different from the 20 per cent alloy.

In Figure 7, illustrating the 40 per cent tin alloy behavior, we see that the prior mechanical history of the material does make a difference. A first loading and a second loading behavior is illustrated in this figure. The second loading is done immediately after the first loading, that is, we load the specimen up to the maximum value, unload it, start reloading it immediately, and you notice there is a decided difference in the amplitude of the count rate.

Figure 8 illustrates the behavior of the 50 per cent tin alloy. Again, we have the same reload representation.
Fig. 3 Acoustic emission in counts per 30 seconds during loading as a function of strain for pure Pb.
Fig. 4 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb -10% Sn.
Fig. 5 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb - 20% Sn.
Fig. 6 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb - 30% Sn.
Fig. 7 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb - 40% Sn.
Fig. 8 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb - 50% Sn.
Figure 9 illustrates the behavior of the 60 per cent tin alloy and now almost all of the emission that we see occurs in the early part of the test. As the slope of the load-displacement curve goes to zero, the emission count rate effectively goes to zero.

Figure 10 illustrates the behavior of the 70 per cent tin alloy. This alloy is very near the eutectic composition. This is the peculiar material which when deformed to 80 per cent deformation becomes superplastic. It should be noted that the scale on the counting rate has been changed by an order of magnitude in this plot.

In Figure 11, illustrating the behavior of the 80 per cent tin alloy, we notice that on the second loading we have picked up a second peak in the emission rate in the early loading part on the second loading.

Figure 12 illustrates the behavior of the 20 per cent tin alloy which is almost in the solid solution region.

Figure 13 illustrates the behavior of pure tin.

Figure 14 illustrates the effect of instant reloading on the behavior of the 40 per cent tin alloy. We have three loading conditions, one right after the other, to show you what the acoustic emission looks like for this particular material, keeping the crosshead speed the same in these three cases. This behavior, when compared with that of the 20 per cent tin alloy shown in Fig. 15 and which was similarly loaded, shows an inconsistency.

In Figure 16 we have endeavored to show the effect of one alloy (50 per cent tin) solidified at three different rates. Going from left to right, the degree of supercooling prior to solidification is increased. The specimen, the behavior of which is shown on the left-hand side, was solidified just about as slowly as we could solidify the material. In the second case we have supercooled it very slightly and solidified it. In the third case we solidified the specimen as fast as we could. In the first instance we had primary crystallization lead solid solution
Fig. 9 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb - 60% Sn.
Fig. 10 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb - 70% Sn.
Fig. 11 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb - 80% Sn.
Fig. 14 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb - 40% Sn with repetitive loading.
Fig. 15 Acoustic emission in counts per 30 seconds during loading as a function of strain for Pb - 20% Sn with repetitive loading
Fig. 16 Acoustic emission in counts per 30 secs. during loading as a function of strain for Pb-50% Sn with varying microstructures.
dendrites which were very small. In the second case we had larger den­
drites of the lead solid solution and in the third case we have very
large dendrites of the lead solid solution. It should be noted that
although the load displacement curve is not appreciably altered by the
microstructural changes, the acoustic emission count rate is drastically
altered.

At this time we have not examined the difference in the emission
produced by fracture, slip, and twinning. In the systems studied, small
deformations do not produce fractures so the bulk of all the emissions
must come from twinning or slip in the specimens. From the data herein
presented we conclude that (1) since the signal from twinning has a
polarity, simple signal amplitudes should not be used in characterizing
the twinning mechanism; and, (2) since microstructure, degree of defor­
mation, prior mechanical history and chemical composition all affect the
emission to a marked degree, analysis of the emission signature cannot
simply characterize the deformation mechanism.