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Triboluminescence from Marble under Uniaxial and Biaxial Stresses

Itaru Maeda

Erimo Geophysical Observatory, Faculty of Science, Hokkaido University, Sapporo 060, Japan

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Abstract

Activity of triboluminescence of marble during sample failure under uniaxial and biaxial stresses was investigated with a time resolution of 100 microseconds. The triboluminescence increases smoothly as the sample failure progresses with increasing speed, then reaches its maximum intensity when the speed of failure is fastest. Up to this moment, the behavior of the triboluminescence doesn't depend on the stress state whether it is uniaxial or biaxial. After the load has gone down to zero, the triboluminescence reduces its intensity linearly in a log-log scale in the uniaxial cases. In the biaxial cases, the situation is not simple because of non-zero external load remained. Three cases occurred according to the amount of the second principal stress. The variation of the triboluminescence shows some irregularities after the time when it attained its peak. Eventually, the intensity decreases linearly in the same sense as the uniaxial cases but the speed of the decrease is faster than that of the uniaxial cases.

1. Introduction

It is not well known, with sufficient resolution of time, of fracture process of rocks, especially after sample failure, though the word sample failure is not defined definitely with the time resolution. In this paper, we will use the word in the following meanings: The beginning of the sample failure is at the time when the load excerted through a ram with constant speed ceases to increase and then begins to fall. The end of the failure is defined by the time when the load becomes zero or ceases to fall depending, respectively, on whether the experiment is uniaxial compression or biaxial one.

One of the methods to examine the process is, along with the usual stress and strain measurements, to measure so called triboluminescence (TL) which is accompanied by crack generation or severe deformation (Walton, 1977). The TL does not accompay any reverberation which acoustic emissions entail and which obscures the subsequent events, mainly, generation of micro-cracks. This characteristic of having no reverberation gives TL of great advantage to be used for the time resolved observations.

TL has another advantageous point : We place the light detectors away from samples, so that we can observe events or phenomena after the sample failure. It is usually very difficult to do this, for example, with acoustic emissions. With this characteristic of TL, Chandra and Zink (1980) were able to examine the fracture process in collision experiments.

In uniaxial fracture experiments, Maeda (1986) reported that TL activity of granite showed complete resemblance with the earthquake activity before and after a large earthquake. In the experiments, the TL after sample failure comes from fragments of the sample just crushed, which are not subjected any external load. There arises a problem of whether the resemblance is universal or not. There are two possibilities that the resemblance is specific to granite which has mineralogical inhomogeneity and that it appears only in a uniaxial experiment in which samples or fragments of samples may not be subjected external load after sample failure.

In this paper, we examine these two possibilities by observing TL of marble, which is mineralogically uniform, in uniaxial and biaxial experiments. In the biaxial experiments, we may expect that the residual load which is exerting after sample failure will not be zero and will give some information on fracture process and on TL behaviour.

2. Experiments

The measuring system for TL and loads is the same as that reported (Maeda, 1986). In the experiments, only the photomultiplier model R943-02 is used to convert TL light quanta to electric pulses which are counted by a so called photon-counter, a pulse counter, with assigned period. The counting period is 100 or 200 microseconds, which determies the time resolution of the measurements. In the biaxial experiments, only TL and one load or only loads in two directions are measured with this time resolution, because a two channel high speed digital memory was available.

The loading machine for the uniaxial experiments is a small uniaxial press the operation of which is to turn a screw with a small piston at one end. Turning the screw constantly, we obtain nearly a constant loading rate. Although, the loading rate depends on the turning speed of the screw in principle, the ratio of the areas of the small piston and larger one which enhances the force excerted to the small piston through pressure oil actually determines the loading rates, because the turning speed cannot be changed over a wide range in actual use. We will call the larger piston a ram.

The same mechanism is employed to construct a machine for biaxil experiments. The machine has four such mechamisms, two in pair and in line with rams opposing with each other and the pairs are allocated in plane and in crossing directions. In actual use, a guide block containing four anvils is inserted into the machine, each four rams of which pushs corresponding one anvil forward, that is, toward the center of the machine. Samples are placed at the center. TL measurements are carried out from the direction perpendicular to the plane of the compressional axes. The ram movement can be controlled with the accuracy of less than 1 micrometer. The maximum power of the machine is 8 tons.

We will call one of the loads the axial load and the other one the side load. In the experiments, both loads are applied to a sample simultaneously in nearly the same rate up to a certain level, then only the axial load is increased while the side load is kept constant.

The samples used in the present experiments are marble the grain size of which is 0.2-0.3 mm. The size of the samples are $4 \times 4 \times 8\ mm^3$ for the uniaxial experiments and $5 \times 5 \times 4\ mm^3$ for the biaxial ones. In the biaxial case, four faces of $4 \times 5\ mm^2$ of the each sample are compressed by the tips of the anvils, each of which have $4 \times 4\ mm^2$ area. Samples are dried at 60°C for more than 12 hours, but experiments are carried out in the so called room conditions.

3. Results and discussion

We begin with the results of the uniaxial experiments. Most of the fractured samples (more than twenty-five out of thirty) showed the same feature which is exemplified in Fig. 1. In accordance with this, patterns of TL activities of most samples are the same and are shown in Fig. 2 and Fig. 3. The sensitivity of light detection is higher in the case of Fig. 2 than of Fig. 3. Under uniaxial compression, sample failure occurs gradually at first, which is demonstrated by the upper traces (showing load variation) in the figures, then the failure progresses rapidly. The speed of the progress reaches its maximum before completion of the failure. We can say, ignoring rapid fluctuation in TL intensity, that the variation of TL intensity is parallel with the speed of the sample failure, that is, the TL intensity grows gradually then attains its maximum when the failure progresses most rapidly.

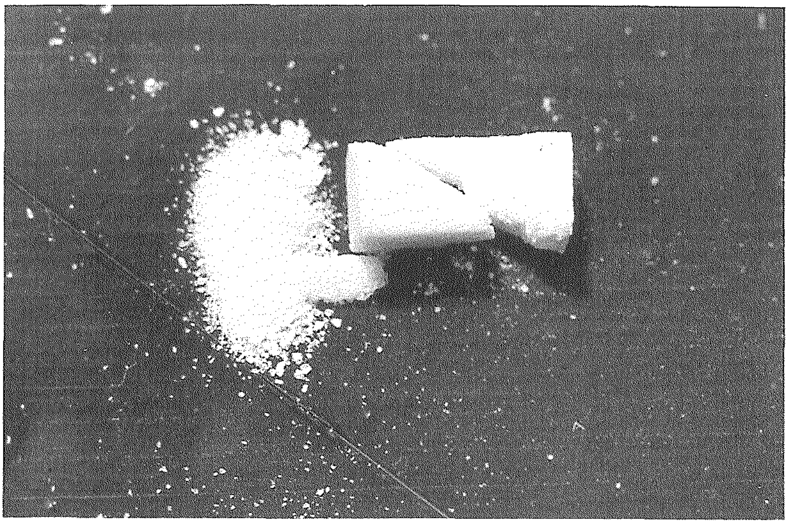

Fig. 1. Crashed sample by uniaxial compression.

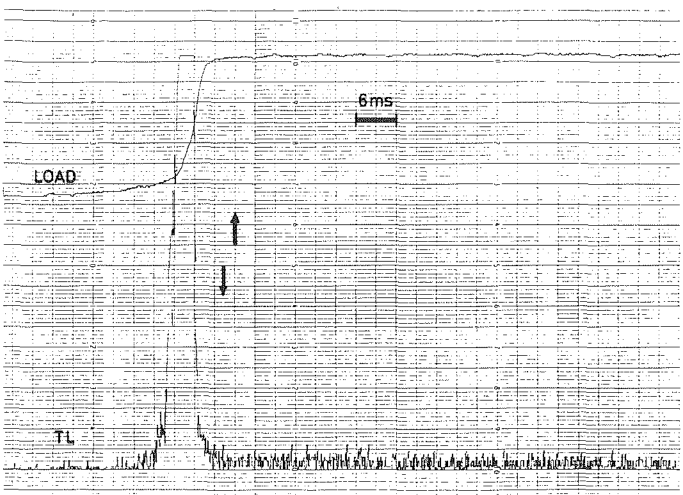

Fig. 2. An example of records of axial load (upper trace) and TL variations obtained in a uniaxial experiment. Relative pen positions for upper and lower traces are indicated by upward and downward arrows respectively. The load trace is presented upside down in the usual sense, so that the uppermost level indicates its zero.

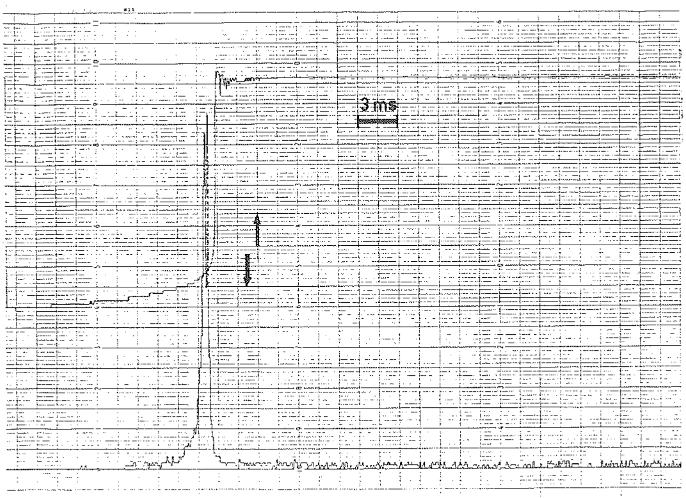

Fig. 3. Another example of records for uniaxial experiments. Refer to Fig. 2 for the meanings of this figure.

The irregular variation, apart from the fluctuation stated above, of TL in growing stage, which is observed for granite, was not observed for all the samples examined. This can be explained by the fact that marble is completely uniform mineralogically, which contrasts to granite. Being uniform mineralogically means uniform efficiency to emit TL and results in a uniform stress

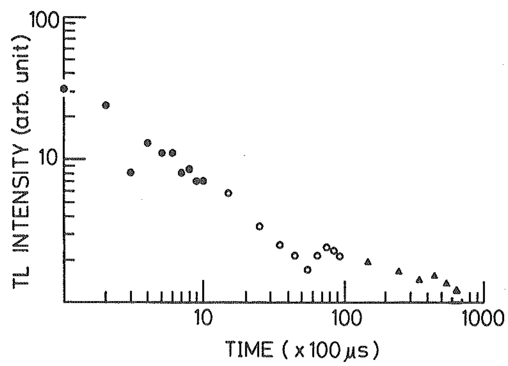

Fig. 4. Variation of TL as a function of time plotted in a log-log scale. The data corresponds to the one given in Fig. 2. The origin of time is when the load reached zero in Fig. 2. Closed circles indicate original values measured, open circles are the averaged ones over ten data, and triangles are the ones averaged over 100 data.

state in an average sense. From these, rather smooth variation can be expected.

Passing the maximum, TL intensity decreases rather rapidly until the load goes to zero, then slowly. The variations plotted in a log-log scale are shown in Fig. 4 and Fig. 5, corresponding to Fig. 2 and Fig. 3 respectively. The origin

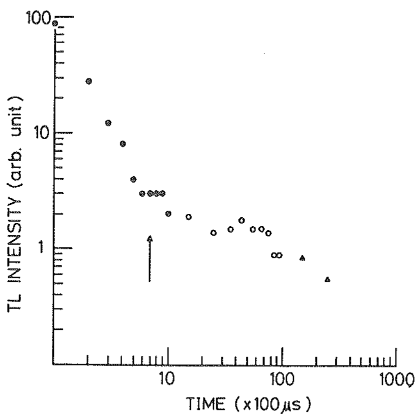

Fig. 5. Variation of TL corresponding to Fig. 3 plotted same way as in Fig. 4 except the origin of time which is the time when TL reached the peak. The moment when load has gone to zero is indicated by an upward arrow.

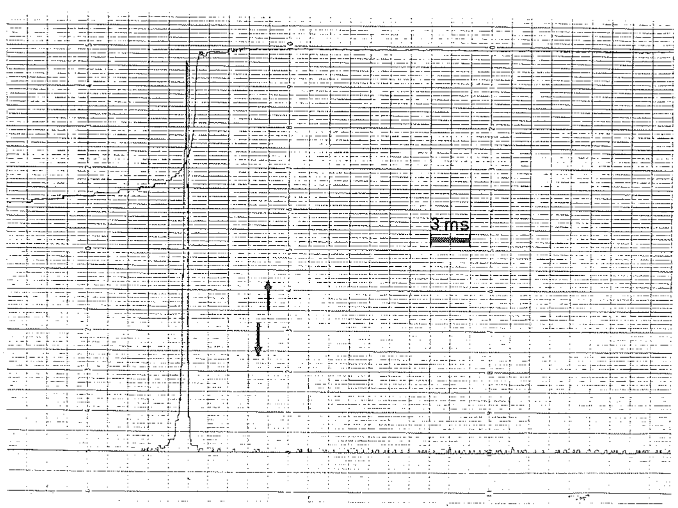

Fig. 6. An exceptional record obtained in a uniaxial experiment. For the meanings of this figure, refer to Fig. 2.

of time in Fig. 4 is the time when the load reached zero and in Fig. 5 it is when TL attained its peak intensity. After the load has reached zero, (in Fig. 5 the time is indicated by an upward arrow) TL intensity decreases nearly linearly in a log-log scale. This behavior of TL is common with the cases of granite and is considered to be an intrinsic feature of fracture process of crystalline aggregates.

The only exceptional case is shown in Fig. 6 in which TL intensity once decreases to a very low level after the load has been removed then seems to recover. This variation can be more clearly observed in Fig. 7 which is plotted in a log-log scale, though the fluctuation is somewhat large because of the low TL intensity. Similar variation is more frequently observed in the biaxial experiments.

We will proceed to the biaxial experiments. We will present the amount of load in terms of stress in a sample. When the side load is sufficiently low comparing to 100 MPa, samples crumble gradually as the axial load increases. The speed of the crumbling is not constant and at some time reaches its muximum. TL glows around this period but its pattern varies from sample to sample. This is completely different from the cases of uniaxial compression and can be explained by the fact that sample shapes and ways of loading are different.

The loading history which is typical in the cases of side load being about 100 MPa is shown in Fig. 8. This figure consists of three parts with different time scales each of which is given in the corresponding part. At sample failure, the axial load drops nearly 100 MPa but the amount of the side load is very little. Before and after the sudden drop, the axial load gradually decreases at nearly

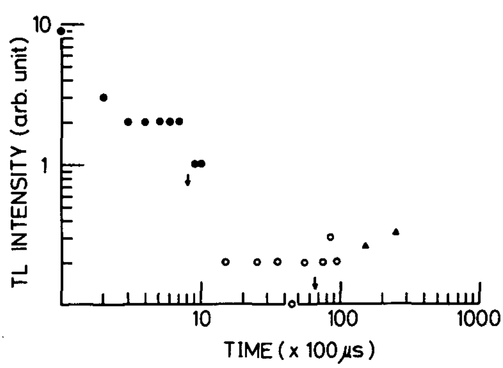

Fig. 7. Variation of TL corresponding to Fig. 6 plotted same way as in Fig. 4

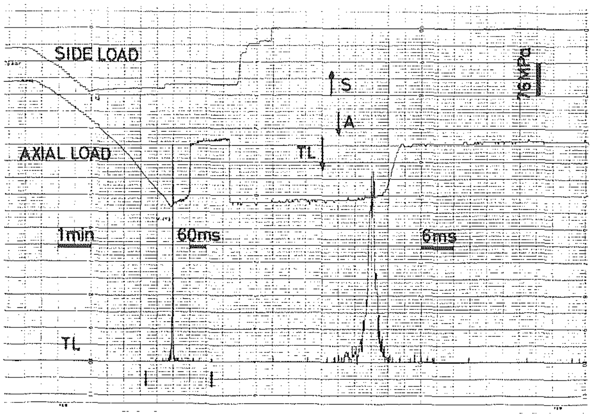

Fig. 8. An example of record for a biaxial case with the side load of 100 MPa. This consists of three parts with different time scales as indicated. Relative pen positions are indicated by small arrows. The upper most trace is the variation of side load, the middle one the axial load, and the lowest one TL. The left most part is a real time record which shows the history of loading. The left most levels of the traces indicate positions of load of 0 MPa. In the middle, variations of axial load and TL are shown in a compressed form and the upper most trace has no meaning. In the right half part, the enlarged record of that given in the middle is shown.

constant rate. The variation of TL intensity seems to be parallel with the speed of decrease in the axial load, which is similar with the uniaxial cases. Unfortunately the sensitivity of the detector for TL is not sufficient to determine whether, after the sudden drop of the axial load, TL intensity decreases linearly in a log-log scale.

In cases of higher side load than 100 MPa, fracture process seems to become complex. During the first stage of failure, as the speed of reduction in the axial load increases, TL intensity increases, then reaches its maximum, which is common to all the cases stated. For some cases, the axial load does not fall to a final level at a stretch but reduces its speed in the middle of the fall. For the other cases, the axial load fall straight to a final level.

We will examine the first cases a bit closely, an example of which is given in Fig. 9. It can be seen from the figure that, corresponding to the step in the variation of the axial load, the variation of TL intensity has a step. By recording the axial and side loads with the same time resolution (see Fig. 10), we can see that, at first, the axial load falls rapidly for nearly 100 MPa but the side load does not, then after the step both loads fall rapidly. It seems that the speed of falling for the side load is faster than for the axial load.

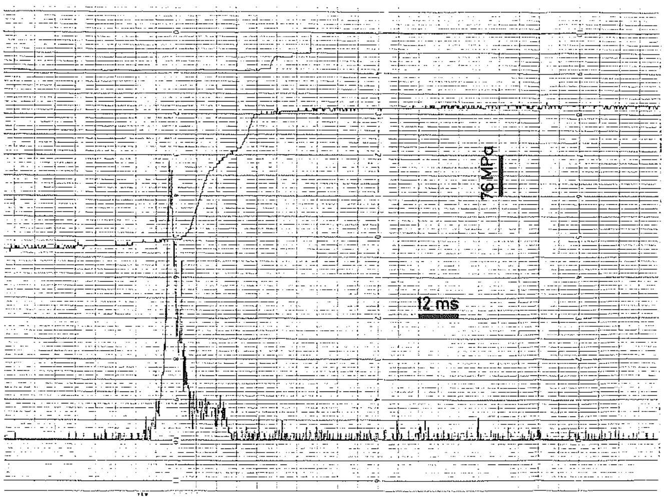

Fig. 9. An example of records obtained when the side load was sufficiently high and having a step during rapid failure.

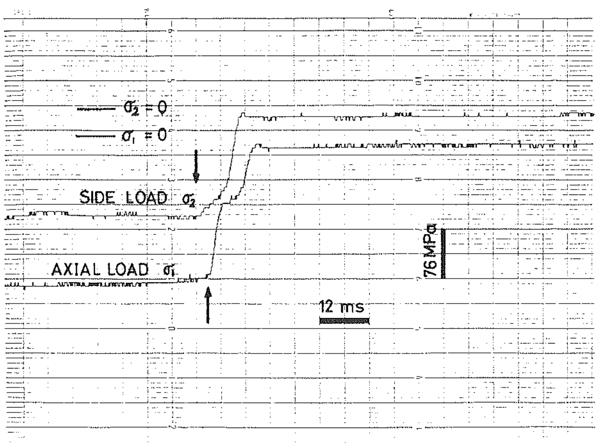

Fig. 10. Time resolved record of axial (lower trace) and side load (upper trace). This shows how each of these loads goes down.

This can be explained as follows: The axial load produces the primary principal stress and the plane of maximum shear may be a plane ABCD in Fig. 11, along which the first fracture will occur. The plane ABCD contains the axis of the side load which results in the secondary principal stress. We can see

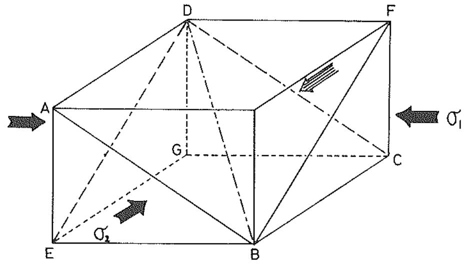

Fig. 11. Possible fracture planes in a sample compressed biaxially and its relationship with axial and side load axes. A chunk DEBCG is expected to be find in the rubble.

that, during the first fracture, the axial stress will fall rapidly but the side load would not. May be because of the impetus of the testing machine, the axial load fall too far and the axis of the primary principal stress turn to coinside with that of the side load. If the stress at this moment remains sufficiently high, second fracture will take place along a plane, for example, DEBF in Fig. 11.

In general, fracture never be confined in a plane, so that as a result of this two step fracture the sample will be crushed completely. If this scenario is true, a chunk of the crashed sample, which is presented by DEBCG in Fig. 11,

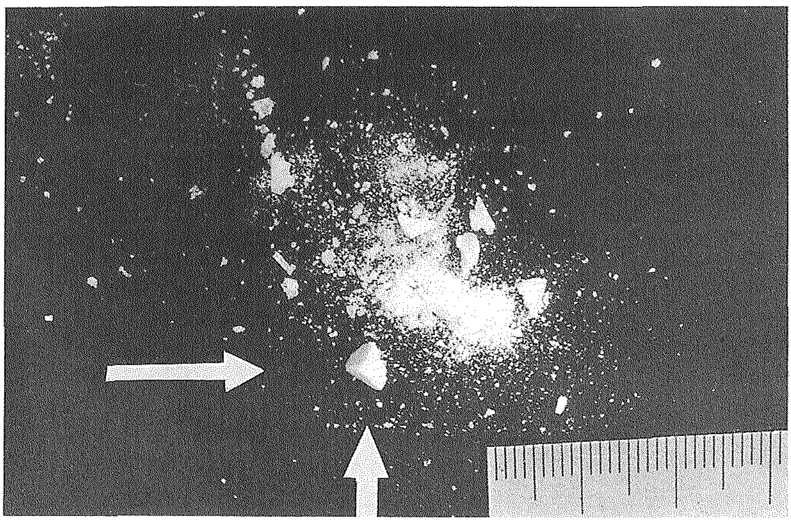

Fig. 12. Fragments of a sample crashed by biaxial compression. A chunk pointed by arrows seems to correspond to the one suggested in Fig. 11

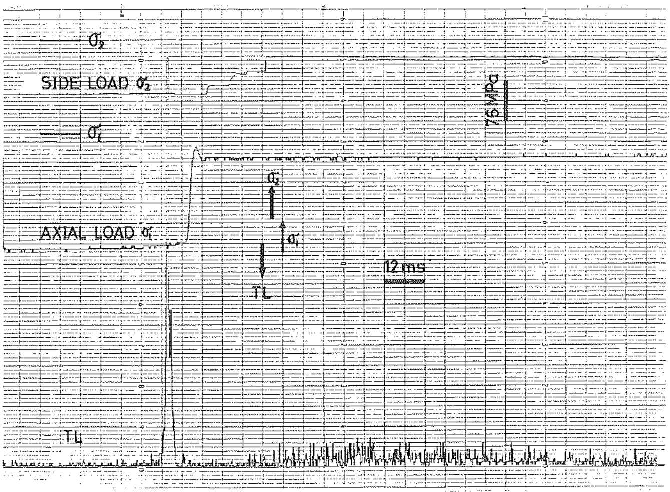

Fig. 13. An example of the rivived axtivity of TL (lowest trace) long after the sample failure.

will be expected. It goes without saying that the chunk must have rough surfaces, jagged edges and rounded corners. This is confirmed by Fig. 12 in which the largest block pointed by arrows seems to correspond to what we are expecting, the chunk. Complete loss of strength by the crush explains the reason why the axial load also falls rapidly during the second fracture.

From the argument given, if the transition from the first fracture to the second occurs continuously, the second cases stated above, a straight fall of the axial load, can be expected. An example of the second cases is given in Fig. 13. Which case will occur depends on the speed of sample failure and the response time of the machine.

After the rapid fall, the axial load decreases very slowly but TL shows strange activity, which is clear in Fig. 13 though it is not in Fig. 9. Fig. 14 shows the activity given in Fig. 13 in a log-log scale. The origin of time of this figure is the moment when the axial load has become nearly constant after the rapid fall. This activity seems to correspond to the case given in Fig. 7. At present, no explanation can be given.

After the peak of this activity (nearly 60 msec after from the first peak), the TL intensity decreases linearly in a log-log scale. That the rate of the decrease in this biaxial case is higher than that in the uniaxial case may be relating to the fact that a residual load is still acting in the biaxilal cases but not in the uniaxial cases, or the fact that fracture strength increases as the side load increases.

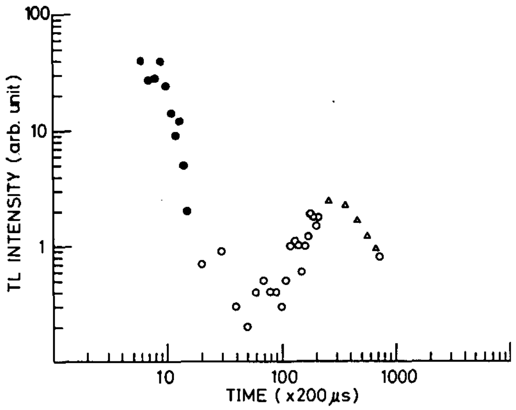

Fig. 14. Variation of TL, corresponding to Fig. 13, plotted in a log-log scale.

The latter means that tougher the rock is, more rapid the decrease of TL is. It should be pointed out, however, that the statement doesn't means that weaker rocks emits TL longer than tougher rocks.

We would point out that, for the marble examined in the present study, the strength of uniaxial compression, the stress drops in the cases of low and middle (100 MPa) side loads, and the stress drops for the first fall in the two step fracture are all nearly 100 MPa. The reason why the value 100 MPa always appears in the experiments carried out under completely different stress states is not known at present.

4. Conclusions

Sample failure begins gradually with low acceleration, which is observed by the speed of load decreasing. The acceleration is rather smooth and the change in TL intensity is parallel to that of the load. No irregular variation in the TL intensity, apart from rapid fluctuation, was observed during this period. This situation is common to both uniaxil and biaxial cases.

Then rapid failure follows. In uniaxial cases, the TL intensity reaches its maximum during the rapid failure, then decreases toward the end of the sample failure. Even after completion of the sample failure, TL is active but decreases linearly in a log-log scale. This is common with the cases of granite.

In biaxial cases, the situation is complex. The situatin is controlled by the side load. The transition from low side lode cases to higher ones seems to be determined by the uniaxial strength of marble examined. Long after (several ten miliseconds) the rapid failure has completed, TL activity revives, then the revived activity decreases linearly in a log-log scale. The speed of the decrease is higher than that of the uniaxial cases.

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