Lifetime-based measurement of mechanical load using mechanical-quenching of CaZnOS:Cu

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The response characteristics of mechanical-quenching (MQ) are discussed and the influencing factors on MQ measurement such as the loading stress, loading rates and loading cycles are analyzed. Moreover, as an evaluation factor, the lifetime is introduced to measure the MQ change, which is proportional to the applied load and is inversely proportional to the loading rate. While the MQ measurement during the multiple loading cycles, the decay lifetime kept stable, indicating that the lifetime can be used for quantitative nondestructive evaluation especially for the multiple loading cycles.

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Mechanoluminescence (ML) is luminescence that is induced by mechanical stimuli,^{1),2)} which offers the advantages of wireless detection, nondestructive analysis, and repeatability. ML materials are promising candidates for applications such as stress sensing and damage diagnosis, particularly for dynamic visualization of stress distributions.^{3)–6)} Recently, we discovered another type of mechano-optical conversion called mechanical quenching (MQ), in which luminescence is quenched by mechanical stimuli.^{7)–9)} In the previous studies, we demonstrated that MQ materials exhibited great potential for visualizing stress distribution, similar to ML. Actually, the response characteristics and mechanisms of ML were fully studied, and the ML measurement system has achieved the practical applications. However, the response characteristics of MQ have not been adequately discussed, and the influencing factors on MQ measurement requires further analysis in order to build the MQ measurement system for quantitative nondestructive evaluation.

On the other hand, the MQ intensity was affected by the decay behavior which is due to the long phosphorescence in our previous report.^{7)–9)} It means the MQ measurement depends not only on the mechanical load, but also the phosphorescence intensity, which increased the difficulty for quantitative nondestructive evaluation.

In this report, we measured the response characteristics of MQ by multiple influencing factors, including the loading stress, loading rates and loading cycles. For better achieve the quantitative nondestructive evaluation of MQ measurement, we also introduced the lifetime as an evaluation factor to analyze the MQ characteristics, because it is typically factor not affected by the phosphorescence intensity.

The CaZnOS:Cu samples were synthesized by a conventional solid-state reaction method. The starting materials CaCO₃ (50 mol % excess), ZnS in stoichiometric ratio and 0.05 mol % Cu₂O were weighed and ground in an agate mortar with ethanol, and then sintered under an Ar flow at 1100°C for 5 h. The excess

calcium compounds were removed from the sample by washing with aqueous acetic acid. After filtering and drying, the samples were ground again and pulverized for measurement.

To evaluate the MQ properties of samples, composite pellets (diameter, 25 mm; thickness, 15 mm) were prepared by mixing the CaZnOS:Cu powders with an optical epoxy resin. The pellet was exposed to UV light (365 nm) for 1 min, left for 3 min, and then the phosphorescence intensity under the load was measured with a lab-built system comprising a universal testing machine (RTC-1310A, Orientec Corp., Tokyo, Japan) and a photon-counting system, which consisted of a photomultiplier tube (R585S, Hamamatsu Photonics, Hamamatsu, Japan), a photon counter (C5410-51, Hamamatsu Photonics), and a computer. The testing machine applied a mechanical load to the pellet, while the photon-counting system detected the phosphorescence. All the MQ intensity (I_{MQ}) has subtracted the phosphorescence intensity.

Figure 1 shows the MQ intensity with the change of the applied load, loading rates and loading cycles. The phosphorescence intensity has been subtracted from the raw data as the background noise. The time, t , was defined to be zero when the load became a maximum. In the Fig. 1(a), the loading rate was fixed at 3 mm/min. The maximum load was varied from 200 to 1000 N. It can be seen that the MQ intensity increased with increasing applied load. Figure 1(b) shows that the MQ intensity with the change of loading rates from 1 to 10 mm/min. The applied load was fixed at 1000 N. The MQ intensity exhibited a rapid growth as the increase of loading rates. It indicates that the MQ intensity of CaZnOS:Cu was affected by the applied load and loading rates, similar to the ML phenomenon. Figure 1(c) shows the MQ intensity under the applied load over five cycles, and the interval between each load was 10 s. The loading rate and maximum load were fixed at 3 mm/min and 1000 N, respectively. It is seen that the MQ intensity shows a slight decrease during the cyclic loading, which is different from some other ML materials.

For better achieve the quantitative nondestructive evaluation of MQ measurement, we introduced the lifetime to further analyze the above MQ properties. The lifetime has been reported to shorten the measurement time and enhance the practicality of ML.^{10),11)}

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Fig. 1. MQ intensity under the different load conditions: (a) under the different applied load (b) under the different loading rate (c) during five repetitions with an interval of 10 s.

Fig. 2. Relationship between the applied load and the influence factors, MQ intensity, recover lifetime τ_{up} and decay lifetime τ_{down} .

Fig. 3. Relationship between the load rates and the influence factors, MQ intensity, recover lifetime τ_{up} and decay lifetime τ_{down} .

The present study focused on the lifetime in MQ measurement. The decay time of MQ, τ_{down} , and the recover time, τ_{up} , were individually calculated using the least squares method:

$$I(t) = Ae^{(-t/\tau)} + C(\tau_{up} : \tau, \tau_{down} : -\tau) \quad (1)$$

Here, $I(t)$ is the MQ intensity at the time t , A and C are constants. As shown in **Fig. 2**, both τ_{down} and τ_{up} were found to increase linearly with increasing applied load. τ_{up} was longer than τ_{down} in all cases. The different time constants indicate that compression and decompression process shows different luminescence mechanism.

The effect of the loading rate was also investigated. The applied load was fixed at 1000 N and the loading rates were changed from 1 to 10 mm/min, corresponding to the Fig. 1(b). **Figure 3** shows the relationship between the MQ intensity and loading rates. The MQ intensity decreased exponentially with the increasing loading rate, which was similar to the reported ML property. Meanwhile, τ_{down} was shown to be shorter than τ_{up} in all cases. The τ_{down} and τ_{up} were found to be inversely proportional to loading rate, and the changing rates of τ_{down} and τ_{up} appear to be nearly equal.

As shown in the Figs. 2 and 3, the decay time τ_{down} and recover time τ_{up} can be fitted very well and show higher accuracy than MQ intensity. Thus, the decay time and the recover time may be useful for evaluating the applied load and loading rate.

Fig. 4. Relationship between the load cycles and the influence factors, MQ intensity, recover lifetime τ_{up} and decay lifetime τ_{down} .

Figure 4 shows the effect of loading cycles on MQ intensity, τ_{down} and τ_{up} . The applied load and loading rate were fixed at 1000 N and 3 mm/min, corresponding to the Fig. 1(c). It could be found that the MQ intensity and τ_{up} decreased with the increase of loading cycles. On the other hand, the decay time

τ_{down} kept stable even with the change of the loading cycles. It indicates that the uncertainty in MQ intensity is still large, but the decay time τ_{down} were not affected by the loading cycles. This behavior is different from the previously reported $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}$, which showed that the lifetime depended on the loading cycles.¹¹⁾ The decreasing MQ intensity of CaZnOS:Cu should be attributed to the continued decline of phosphorescence intensity, and the independent lifetime of CaZnOS:Cu is probably due to its steady nonradiative recombination process.^{7)–9)} However, the physical meaning of lifetime in MQ process is not yet clear. Future studies are required to clarify its mechanism in detail.

In the summary, this report discussed response characteristics of MQ, analyzed the influencing factors on MQ property and first focused on the time constants, τ_{down} and τ_{up} on the MQ measurement. The decay lifetime τ_{down} and recover lifetime τ_{up} are proportional to the applied load and is inversely proportional to the loading rate. Meanwhile, the measured decay lifetime τ_{down} is independent of the loading cycles. There is a possibility that the applied load and loading rate could be quantitative nondestructive evaluated without the influence of the loading cycles.

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