Evaluation of Two-part Epoxy Adhesive using a Rigid Body Pendulum Instrument

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■ Introduction:

I would like to share an incident that occurred within A&D. In the manufacturing process of a product, a two-part epoxy adhesive was used, but there were issues with the adhesive's unstable quality. Although I cannot disclose which adhesive product was used, it was applied in the sensor unit. To better understand the situation, I visited the manufacturing department to see how it was being used in practice. I thought that a rigid body pendulum instrument would be helpful to assess the situation.

■ Problem:

1) Suspected improper curing process.

After manufacturing, the performance of the product does not stabilize for a long period of time.

Advice:

The adhesive was being prepared in a container that was also used to mix the two parts together, and there were remnants from the previous use left inside.

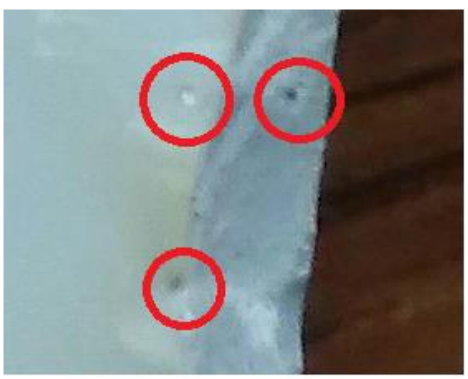
⇒ Cured and uncured parts may mix together.

⇒ Containers should be used only once and then disposed of.

2) The volume shrinks after filling. Can more adhesive be added afterwards?

⇒ It is unclear what will happen at the interface where the newly added adhesive meets the old adhesive.

3) The cut surface after curing shows vacancy (marked with a red circle).



■ 1. Adhesive curing property measurement

Since it seems to be from a major manufacturer and has been used for a long time, I thought that there would be a correct "usage guide". I thought it would be necessary to determine the curing temperature and curing time, which are also features of the rigid body pendulum instrument, and determine the curing temperature and curing time using the ISO12013-1 method.

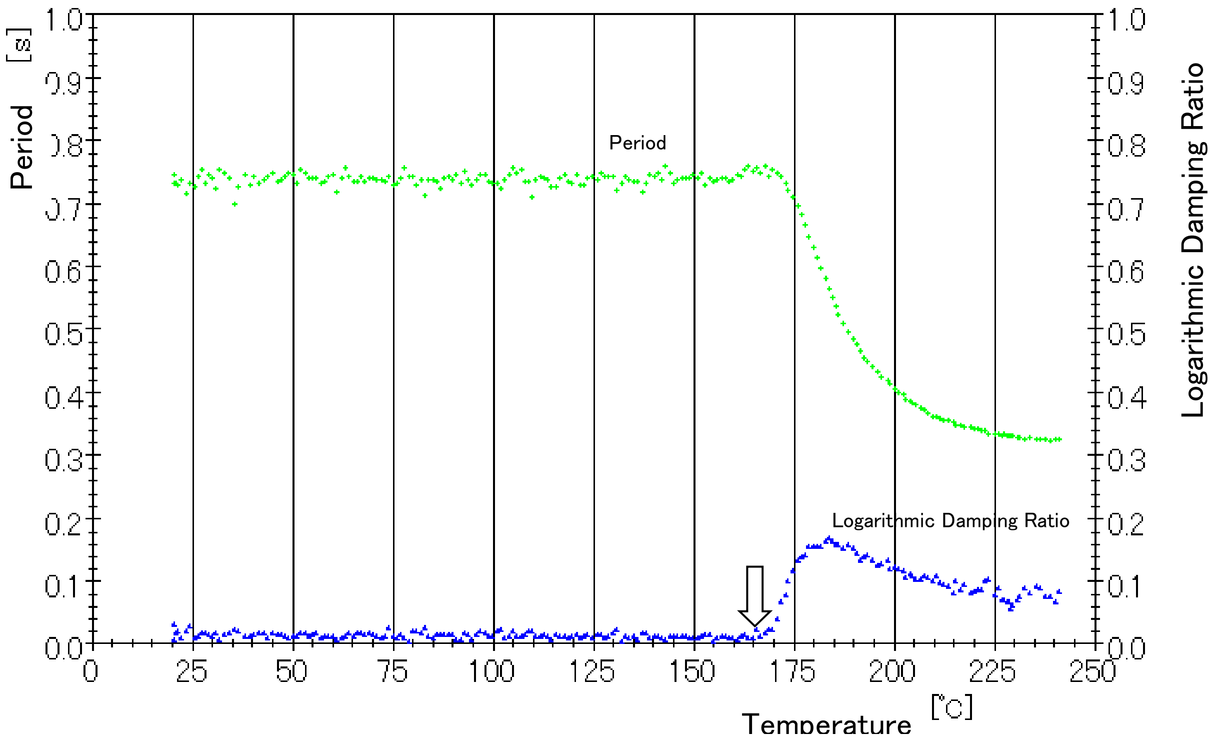


Fig. 1 The method used to determine the curing temperature in accordance with ISO 12013-1.

Curing agent: ****** Co., Ltd. Resin: ****** Co., Ltd.
Mixing weight ratio of curing agent to resin: 0.15/1
Heating rate: 10℃/min
Substrate: SUS (coating thickness of 200 μm)
Pendulum: FRB-****** Edge: RBE-******
Equipment used : RPT-3000W (A&D)

※ The curing temperature was determined to be 165℃ (indicated by the arrow) based on the onset temperature of logarithmic decrement.

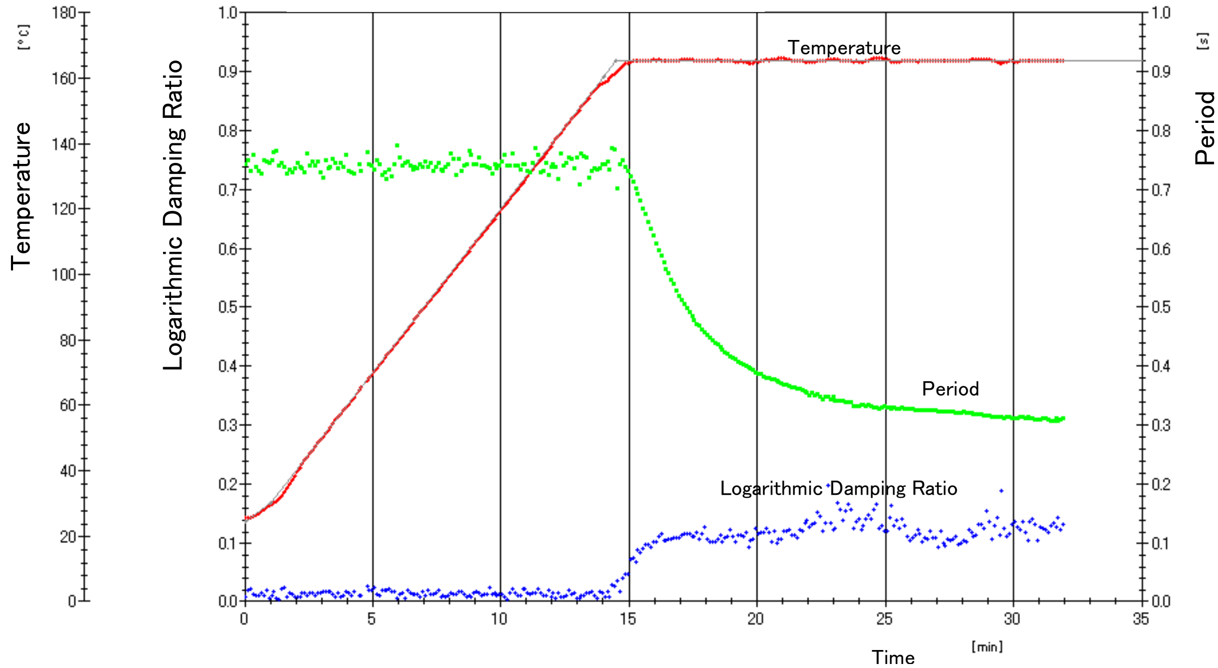


Fig. 2　Hardening time (held at 165℃) test (≒35 mins)
※ It takes about 30 minutes or more for the period to stabilize.
As a basic characteristic of the material, it is OK at around 165℃/35 mins.

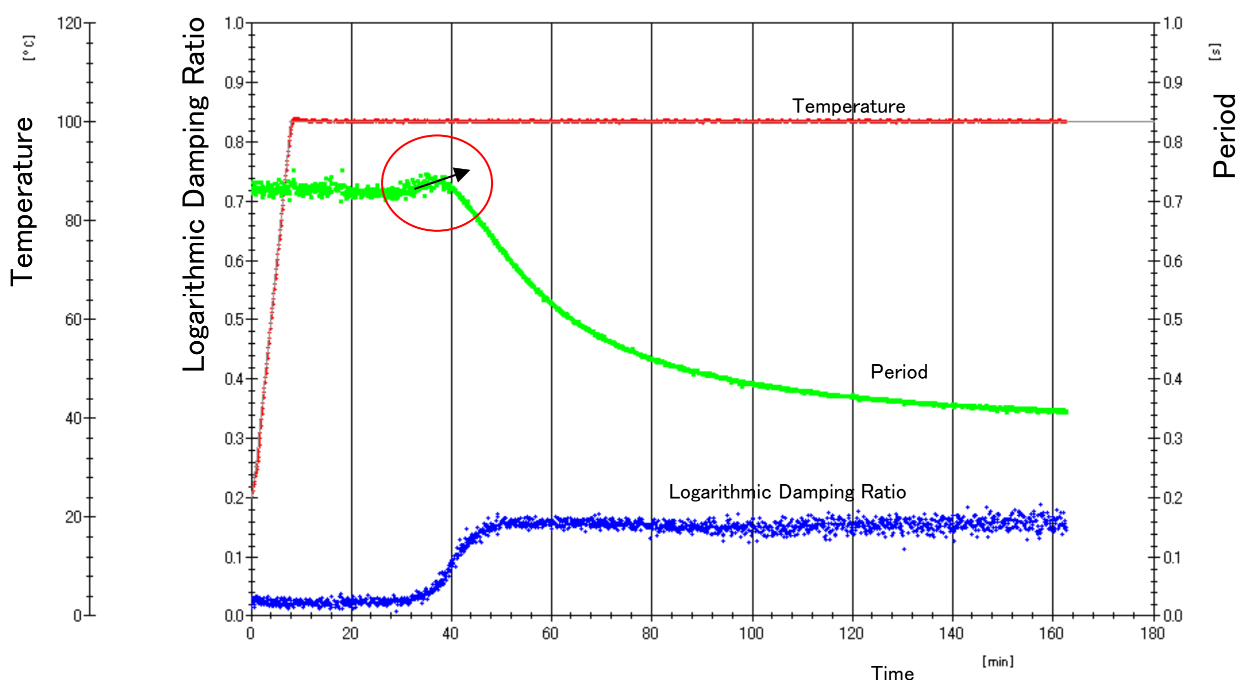


Fig. 3　Curing characteristics at 100° C

Requires at least 3 hours for Period stabilization

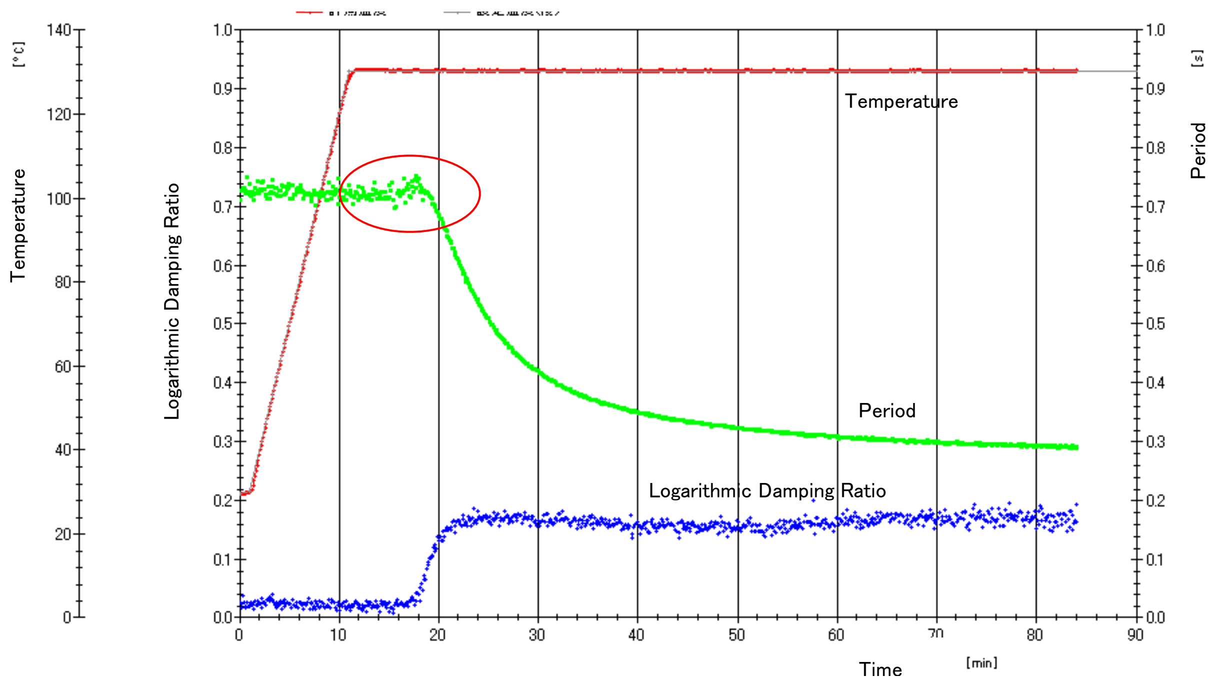


Fig. 4 Curing characteristics at 130° C
Requires at least 1.5 hours for Period stabilization

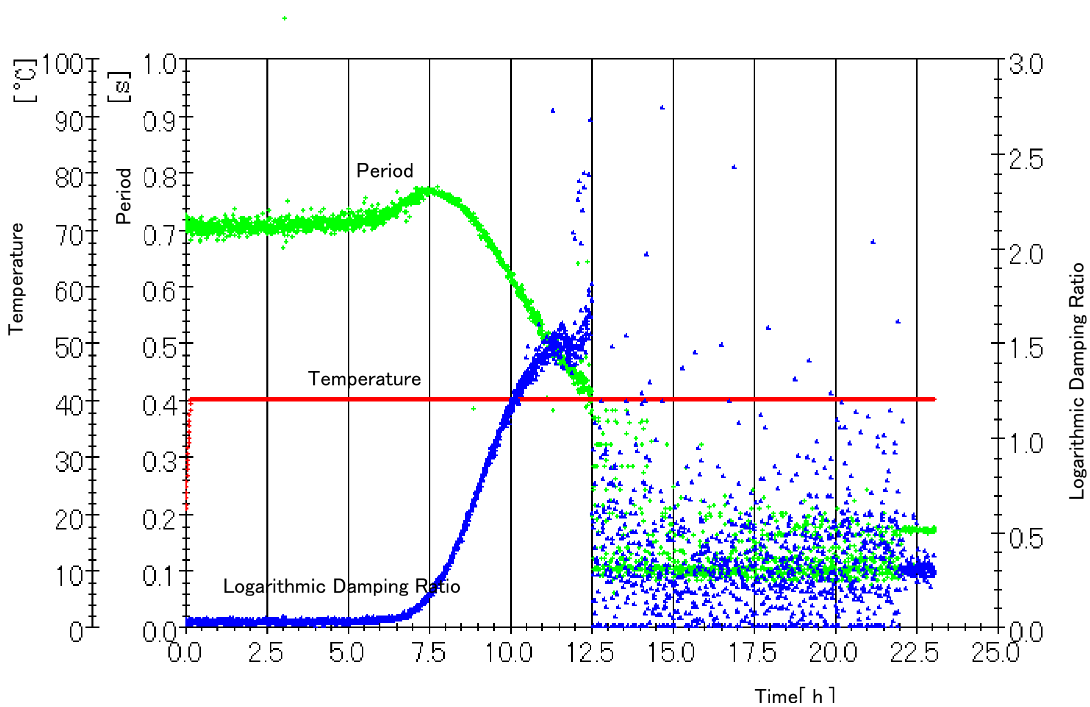


Fig. 5 Curing characteristics at 40 ℃ (ID6663) Requires at least 23 hours

※ The fluctuations in data between 12.5 - 22.5 hours are due to the damping vibration being close to the critical damping vibration.

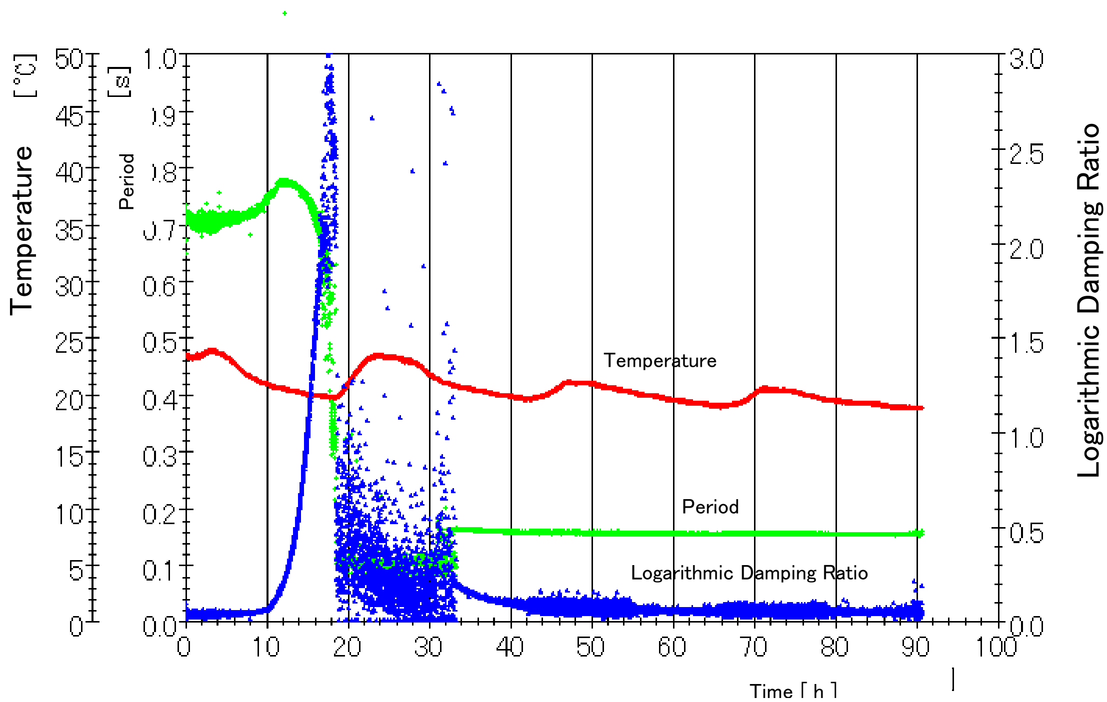


Fig. 6 Curing test (ID6661) at room temperature takes 45 hours.
※ The fluctuations in data between 18 – 33 hours are due to the damping vibration being close to the critical damping vibration.

■ Summary of Curing Temperature and Curing Time

Curing Temperature (℃)	1000 × (1/T)	Curing Time(H)
165	2.28	0.7
130	2.48	1.5
100	2.67	3
40	3.19	23
22 (RT)	3.39	45

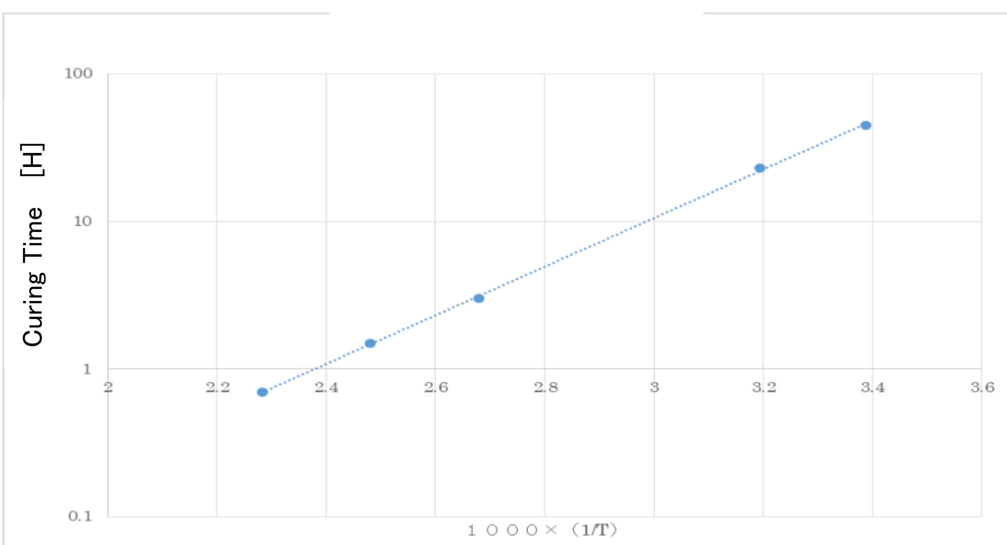


Fig. 7 Relationship between curing temperature and curing time

■ Continuous measurement of properties (ISO12013-2) from curing (ISO12013-1)

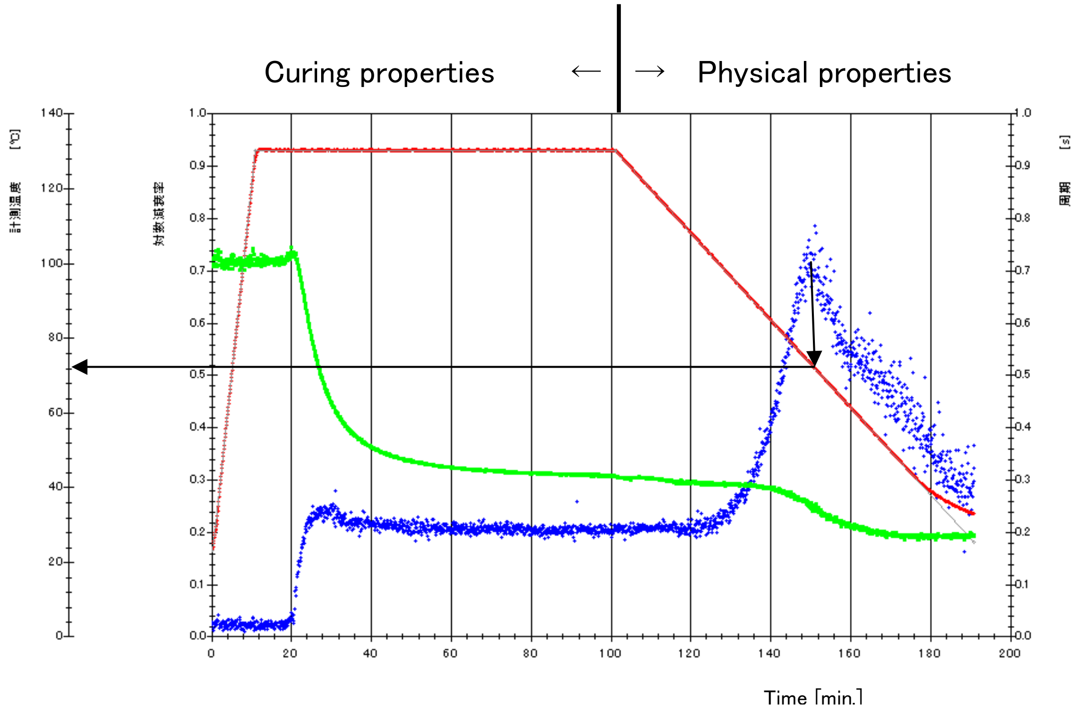


Fig. 8　The physical property measurement (Tg(ISO12013-2) ≒ 73℃) after curing.

※ Example of test specimen (after measurement)

Fig. 9　Sample condition after measurement

■ Results and Discussion:

1. The relationship between curing temperature and curing time was obtained.
2. The graph shows some irregularities in the period, which is not commonly observed in uniform materials. This may be due to the incorporation of bubbles during mixing of the two liquids, which gradually come out after coating, resulting in a decrease in the thickness of the sample. The fact that the period values are already irregular at the beginning of the measurement also supports this hypothesis.
3. The reason why the red circle part is formed is probably because the air bubbles did not come out and it hardened. Unfortunately, we have not been able to examine the conditions under which the particles are not included in the cured product. However, it can be expected because it seems to be captured as a phenomenon by measuring with a rigid body pendulum type instrument.
4. The glass transition temperature (Tg) was determined without detaching the coating.