Compressive deformation of expanded polylactic acid resin subjected to quasi-static and dynamic loadings at wide temperatures

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Abstract
Many kinds of polymer foams are used in various situations. Especially, polymer foams are often used for shock absorber, therefore, there are many studies focusing on the effect of strain rate on the compressive properties, such as strength and absorbed energy. Since, most of them is produced from petroleum, they are not so environmentally friendly. Recent years, therefore, polylactic acid resin foam was developed that is a kind of plant-derived plastics with low environmental load. In this study, the effect of strain rate and temperature on the compressive property of polylactic acid resin foam was experimentally studied by carrying out the compression tests at various strain rates from 0.001 to 790 s⁻¹. It was found that the flow stress of polylactic acid resin foam during compressive deformation increases with the increase of strain rates. It was also found that the flow stress of polylactic acid resin foam decreases with the increase of temperature. In addition, polylactic acid resin foam indicates brittle behavior near liquid nitrogen temperature and impact compression caused greater effects of brittleness than quasi-static tests.

Key words: Polylactic acid resin foam, Strain rate, Temperature, Impact, Embrittlement

1. Introduction
These days, many kinds of polymer foams are used for shock absorber or thermal insulator in various daily situations because of their characteristic mechanical properties. Therefore, there are many studies, (Bouix, et al., 2009, Ouellet, et al., 2006, Subhash, et al., 2006, Sorrentino, et al., 2007, Viot, et al., 2005) on the constitutive equations and impact energy absorption of polymer foams using Split Hopkinson Pressure Bar (SHPB). Most of the polymer foams are produced from petroleum. In general, petroleum products lead to an increase in carbon dioxide emissions, which is undesirable because it is said to be one of reasons for global heating. Recently, polylactic acid resin (PLA) produced from plants are attracting attention as a material that gives little damage on environment because of its carbon neutrality, (Ema, et al., 2006, Hagen, 2012). PLA is a relatively new polymer material, it is used for the body of the electric apparatus or foams (Expanded PLA : EPLA). Conventional EPLA is poor of dimensional stability at high temperature (over 60 °C), thus its use has been limited. By the improvement in the manufacturing processes, however, it becomes possible to develop a new EPLA that has good dimensional stability at 150 °C. This new EPLA is a possible alternative to the expanded polystyrene (EPS), it has been expected for using as the automobile parts or thermal insulator. However, the effects of strain rate as well as temperature on the compressive property of EPLA have not been fully evaluated. Therefore, it is quite important and useful to clarify its mechanical property in order to encourage the dissemination of materials that provide small environmental load.

In this study, the effects of strain rate and temperature on the constitutive relation of a newly developed EPLA were experimentally examined by performing a series of compression tests at wide range of strain rate (1.1×10⁻³ ~ 7.0×10² s⁻¹) and at various ambient temperatures.
2. Experiment
2.1 Materials and Specimen

EPLA specimen was produced by cutting an EPLA plate with a thickness of 30 mm provided by Sekisui Plastics Co., Ltd.. Specimen is a cylinder with a diameter of 30 mm, and a height of 30 mm as shown in Fig. 1. We prepared two kinds of specimen with different density of 60 and 200 kg/m$^3$. They have two different foaming magnifications of 20 and 6, denoted by EPLA-A and EPLA-B, respectively. These foams have closed-cell structure. The EPLA specimens in this study have individual differences in the density about ±20 % and the average of density of 90 specimens are 63.5 and 201 kg/m$^3$ respectively, which almost coincide with the catalogue values, shown above. Therefore, we needed to cancel the effect of density variability to increase the accuracy of experimental data.

2.2 Compression Tests

By using a screw-gear type universal testing machine (Shimazu Autograph), quasi-static compression tests were carried out at strain rates of 1.1×10$^{-3}$ and 1.1×10$^{-1}$ s$^{-1}$. Specimens were compressed up to a strain of about 0.8. In high temperature tests of 70 °C and 120 °C, specimens were pre-heated at these temperatures for 15 minutes in electric furnace to ensure that the temperature becomes uniform in the whole specimen body. These temperature were chosen not to exceed the melting point $T_m$ (= 169 °C) of this EPLA. In low temperature tests, specimens were similarly pre-cooled to -190 °C for 15 minutes in a thermostatic bath (polystyrene foam box) as schematically shown in Fig. 2. The specimens are just cooled by the heat conduction of upper and lower anvils and cold atmosphere.

A dropping weight testing machine, shown in Fig. 3, was used for intermediate speed compression tests. The strain rate is approximately 10 s$^{-1}$. The deformation of specimen was measured by a high speed camera (Phantom V4.2). The load was measured by a special load cell made of duralumin. It consists of a small detective section and a large stress-transmitted block. Similar method to detect the load was often adopted in dynamic tests which requires a relatively long time for measuring load, (Tanimura, et al., 2002).

The direct impact compression test apparatus is shown in Fig. 4. A striker that accelerated by compressed air, impacts the specimen attached to the special load cell. Its strain rate is about 700~800 s$^{-1}$. The load and displacement were measured by the same method as that used in the dropping weight tests.

Impact tests were also carried out at high and low temperatures which were adopted to be 83 K (-190 °C), 343 K (70 °C) and 393 K (120 °C) as well as at room temperature. When high and low temperature impact tests are performed, a little ingenuity is required because the small detective section of load cell (mentioned above) is affected by heat. It is not appropriate that the pre-heated or pre-cooled specimen is attached to the load cell directly. Therefore, the specimens attached to the striker, not to the load cell, was pre-heated by the electric furnace or pre-cooled. The setup used for high temperature impact tests is shown in Fig. 5. As a result, heated specimen was compressed at impact strain rate without thermal effect to the load cell. In order to obtain an enough acceleration distance for the impact velocity, a long striker was adopted. In low temperature tests, it was difficult to attach the thermostatic bath to this apparatus. Therefore, the specimen was pre-cooled by the thermostatic bath located at just neighbor place, then we attached it to the load cell and compressed immediately after taking it out from the thermostatic bath.
3. Experimental Results and Discussions

3.1 Density Normalization of Specimen

Generally, most foam materials have the distribution of density because of the variety of cell size. Therefore, the specimens made from such foam materials involve individual difference in the density and this difference causes the scatter in the mechanical properties of specimens measured, (Gibson and Asby, 1999). Therefore, it is quite important to eliminate the effect of specimen density in investigating the mechanical properties of foam materials. For this purpose, we defined two ratios: one is the ratio of specimen density, \( \rho^* = \frac{\rho_1}{\rho_2} \), and the other is the ratio of representative stress, \( \sigma^* = \frac{\sigma_1}{\sigma_2} \), where the representative stress was defined as the mean stress in the range of strain from 0.2 to 0.4. The subscripts of 1 and 2 indicate two arbitrary specimens tested under the same condition, thus, if we have 4 specimen data, we can plot 12 points (4×3) on a log-log scale graph as shown in Fig. 6. In this figure, 32 specimen data of EPLA-A and -B tested at room temperature are involved. From this graph we determined the equation expressing the relationship between \( \rho^* \) and \( \sigma^* \) as follows,

\[
\sigma^* = \rho^{n^*}
\]

When \( n^* \) is the density sensitive exponent given by the slope of a fitting line in the figure. We drew 10 figures (4 figures : at room temperature, 4 strain rates, 6 figures : at the three temperatures, 2 train rates) and obtained an average
values of $n^*$ of 1.62 because all $n^*$ values scattered from 1.58 to 1.66. All experimental data were normalized by applying this equation to be the density of 60 kg/m$^3$ for EPLA-A and 200 kg/m$^3$ for EPLA-B, respectively. By this normalization, the effect of individual difference in the density of specimen was almost eliminated. All discussions shown below are based on these normalized data.

### 3.2 Strain Rate Dependence

The stress-strain curves of EPLA-A and -B obtained from compression tests at various strain rates at room temperature (R.T.) are shown in Fig. 7. Generally, compressive behaviour of polymer foam consists of three stages, i.e. linear elastic, plateau and densification regions. Both of EPLA-A and -B indicate clear elastic region, not plateau but gradually increasing region and densification regions as seen in Fig. 7. In both EPLA-A and -B, the increase of strain rate caused the increasing of flow stress at wide strain rate range. Therefore, it can be said that our EPLA foams show clear strain rate dependence from the quasi-static strain rate ($\dot{\varepsilon} \approx 1.1 \times 10^{-3}$ s$^{-1}$) to the impact strain rate ($\dot{\varepsilon} \approx 7.0$–$8.0 \times 10^2$ s$^{-1}$). One of the principal causes of this strain rate dependence can be considered to be the strain rate dependence of mechanical property of PLA itself, which has already been reported, (Tanaka, et al., 2005) although the PLA used is not necessarily to be exactly same as our PLA material. Another reason of the dependence may be the effect of foam structure. When a foam material is compressed dynamically, there is not enough time for the gas inside foams to flow out of foams. Thereby, the pressure of the air inside foams rises and its affects as the resistance of compressive deformation. From these considerations, therefore, we can say that the strain rate dependence of compressive property of EPLA is principally caused by these two factors, i.e. the rate dependence of PLA and closed foam structure.

In order to confirm the effect of strain rate in more detail, the flow stress at each strain $\varepsilon = 0.1, 0.3, 0.5$ were plotted against strain rate, as shown in Fig. 8. The vertical axis of these graphs is taken on a log scale. From these graphs, clear
strain rate dependence can be confirmed at wide strain rate range. We can see that the flow stress increases with increasing of strain rate at a constant rate. And, it can be also confirmed that the gradient is always constant regardless of the different strains. We calculated this gradient $m$, and obtained $m = 0.035$ for EPLA-A and $0.045$ for EPLA-B, respectively. This gradient $m$ corresponds to the strain rate sensitivity exponent.

### 3.3 Temperature Dependence

The stress-strain curves of EPLA-A and -B obtained from quasi-static tests ($\dot{\varepsilon} \approx 1.1 \times 10^{-3} \text{ s}^{-1}$) at various temperatures from 83 K to 393 K are shown in Fig. 9. It is clearly found that the flow stress decreases dramatically with the increase of temperature. This means that the compressive strength of EPLA has great temperature dependence. In the stress-strain curves obtained from higher temperature tests of 343 K and 393 K, the first rise in the elastic region is extremely small and just ordinary rise of stress appears in compaction region. In the results of low temperature tests of 83 K, however, the flow stress is much greater than that obtained from R.T. tests. Therefore, it seems that PLA becomes harder and probably more brittle at low temperature. These tendency can be considered to be the reflection of PLA’s mechanical properties, (Kosugi, et al., 2008). The curve shape of EPLA-B tested at 83 K is very different from that at R.T., i.e. very steep slope is observed from $\varepsilon = 0.05$ to $\varepsilon = 0.4$. The density of EPLA-B is about 3 times greater than that of EPLA-A. Thus, we may consider that the property of PLA itself appeared more remarkably on the mechanical behaviour of EPLA-B.

The flow stresses at strains of $\varepsilon = 0.1, 0.3, 0.5$ were found from the test results and plotted against temperature, as shown in Fig. 10. From these results, we can see that the flow stress is very sensitive to temperature in the elevated temperature range. This is because for both EPLA materials, the temperature increase of about 50 K from R.T. brings
the dramatic decrease of flow stress i.e. they decrease to be almost a quarter of that at R.T. Although, the flow stress goes up to be about twice of stress at R.T. in the low temperature tests, the former decrease of flow stress due to temperature rise is severer than the latter increase in the low temperature range.

The stress-strain curves obtained from impact tests ($\dot{\varepsilon} \approx 400 \text{s}^{-1}$) for EPLA-A and -B at various temperatures from 83 K (-190 °C) to 393 K (120 °C) are shown in Fig. 11. However, because of the capability of direct impact apparatus, we could not compress EPLA-B to the densification region at 83 K. In the same way as the static results, the flow stress at each strain $\varepsilon = 0.1, 0.3, 0.5$ for each temperature are shown in Fig. 12. As well as the quasi-static data of both EPLA-A and -B, the flow stress decreases significantly with the increase of temperature in the elevated temperature range. However, the increase of flow stress between R.T. and 83 K is much smaller than that observed in quasi-static data. Therefore, it is found that in the low temperature range, the effect of temperature during impact compression is smaller than that obtained from quasi-static compression tests. In addition, at room temperature, the flow stress at each strain $\varepsilon = 0.1, 0.3, 0.5$ obtained from impact tests are greater than quasi-static results. However, at 83 K, the flow stress obtained from impact tests is similar to or lower than the quasi-static results. This is because the failure due to brittleness of PLA at 83 K occurred continuously during impact test i.e. it is considered that the increase of flow stress due to the low temperature has been offset by the low-temperature brittleness of PLA strengthened by dynamic testing speed. This brittleness of EPLA can be confirmed from Fig. 13, which shows the specimen after tests at 83 K and room temperature. The specimen tested at room temperature only shortens in height as shown in Fig. 13 (a). In the other figures (see Fig. 13 (b), (c)), however, both specimens are fractured. In the specimen tested at quasi-static rate and at
83 K, circumferential cracks are produced near side surface as shown in Fig. 13 (b). After impact compression at 83 K, the specimen is broken into many pieces. At 83 K, therefore, we can say that impact compression causes severe damage to the specimens.

4. Conclusion

The effects of strain rate and temperature on the compressive property of EPLA were investigated by a series of compression tests. The principal results obtained are as follows:

1) The flow stress during compressive deformation of EPLA shows clear positive strain rate dependence at wide strain rate range from $1.1 \times 10^{-3}$ s$^{-1}$ to $7.9 \times 10^{2}$ s$^{-1}$. This strain rate dependence is considered to be caused by the strain rate dependence of the mechanical property of PLA and the effect of microstructure of foam materials.

2) The flow stress during compressive deformation of EPLA indicates significant temperature dependence and decreases with the increase of temperature, although this is limited in the range between 83 K and 393 K.

3) At 83 K, EPLA becomes brittle due to the mechanical property of PLA itself. The effect of the brittleness of EPLA on the stress-strain behaviour at 83 K appears especially large in the results of impact tests.

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References


