Food Sci. Technol., Int., **1** (1), 26–28, 1995

Effect of Lipid on the Compressive Fracture Stress of Concentrated Amorphous Solution in Frozen Foods

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Received April 12, 1995

The compressive fracture stress of lipid-free tofu was measured at temperatures between −20°C and −196°C and compared with that of lipid-containing tofu to examine the effect of lipid on the fracture stress of concentrated amorphous solution (CAS). In the calculation of the fracture stress of the CAS, the compressive fracture stress of food was regarded as a volume-fraction-weighted average of the fracture stress of each component of which the food is composed. The fracture stress value of CAS in frozen full-fat tofu was found to agree with that of lipid-free tofu provided that the frozen tofu was assumed to consist of pure water ice, lipid, and lipid-free CAS. The compressive fracture stress of selected frozen foods other than tofu was also measured.

Keywords: fracture stress, yield stress, tofu, lipid, CAS

Although very few papers have been published on the mechanical properties of frozen food, the need for knowledge of the mechanical properties of frozen food is expanding such as for an appropriate procedure for spraying liquid nitrogen to avoid crack formation, for a cryo-mechanical process for separation of low fat meat from fatty meat (Hagura & Watanabe, 1991), for comminution in the manufacture of reformed and restructured meat products (Dobraszczyk *et al.*, 1987), and for cutting a large block of frozen tuna meat into pieces of reduced size appropriate for retail (Okamoto *et al.*, 1994). On the other hand, mechanical techniques are recognized as particularly suited to studying the molecular motions that give rise to the glass transition temperature (T_g) and relaxation below T_g (Wetton, 1984).

In their previous paper, Watanabe *et al.* (1995a) measured the compressive fracture stress of frozen tofu (a soy-protein jelly food), which was selected as the sample because its moisture content was easy to vary. They analyzed the data by a simple two-component model consisting of pure water ice and concentrated amorphous solution (CAS), and obtained the fracture stress of CAS, which was a unique function of temperature. However, there is room for improvement in their paper. One point is that the CAS in their paper contained a considerable amount of lipid because they used tofu made of full-fat soybean (ca. 20% wt lipid, 12% wt moisture). Therefore, the effect of lipid on the fracture stress of CAS is required to be known.

Another criticism may concern the maximal freeze-concentration, C_g', of tofu which was assumed to be 100% in their paper. There is a possibility that glass transition may take place when tofu is cooled down to −80°C. In this situation, the maximal ice content in frozen tofu may be lowered and hence the calculated values for fracture stress of CAS may also be lowered. Unfortunately, however, the maximal freeze-concentration, C_g', for soy-protein in tofu has not been known yet. Thus in the present paper, C_g' was assumed to be 100%, and the study was confined to the effect of lipid on the fracture stress of CAS. A discussion concerning the fracture stress of moisture-containing CAS of food in a glassy state was given elsewhere (Watanabe *et al.*, 1995b).

Materials and Methods

The samples used were lipid-free tofu, "Surimi," raw cod fish meat, wheat flour dough, egg white and agar jelly.

Lipid-free tofu was prepared from defatted soy flour (2.5 wt% lipid, 1.4 wt% moisture). Defatted soy flour was suspended and heated to dissolve it in water, filtered through cotton cloth, and calcium sulfate was added to the filtrate to coagulate the soy-protein. The soy-protein was then separated from the supernatant and dehydrated by pressing in a frame.

"Surimi," minced raw fish meat of Alaska pollack containing food additives (5–7 wt% sugar and 0.2 wt% polyphosphate), was purchased in the market and used as is. The cod meat purchased in the market was minced by a knife and crushed in a mortar. Wheat flour and agar powder purchased in the market were mixed separately with distilled water to prepare wheat flour dough and agar jelly. Egg white was separated from hen eggs purchased in the market.

Tofu and each of the other materials described above were separately pressed into aluminum tubes (10 mm i.d., 20 mm long) and were frozen in a freezer kept at −30°C for 12 h. Cylinders removed from the tubes were used as specimens for compression.

The testing machine and the procedure used were the same as those described elsewhere (Watanabe *et al.*, 1995a). In the compression tests, the contact area between the specimen and the bearing plate considerably affects the results of measurement (Tang *et al.*, 1994). When a specimen's surface is rough, it is forced to receive the load on a reduced surface of contact which causes fracture at an apparently lower load than that when the surface is smooth. Smooth contact is often achieved by "ice capping" which involves placing a small amount of cold water into the thin clearance on the specimen; the water soon freezes and the contacting surface is smoothed (Hagura, 1991). When we applied "ice capping" in this study, a pair of heads (aluminum disks 8 mm id. 5 mm thick) was inserted between the specimen and the bearing plate in order to make it easy to apply "ice capping" to the upper end of the specimen (Tang *et al.*, 1994).

Results and Discussion

The stress-strain curves in the compression test on lipid-free samples under a constant rate of deformation showed ductile fracture in the range between −20°C and about −80°C, while they showed elastic deformation followed by a sudden fracture in the range between about −80°C and −196°C.

The compressive fracture stress, which in this paper refers to the maximum stress in a stress-strain curve, is plotted against temperature for lipid-free tofu in Fig. 1, which looks nearly the same as that for lipid-containing tofu (Fig. 4 in the previous paper: Watanabe *et al.*, 1995a) except that (a) the fracture stress of lipid-free tofu was larger than that of full-fat tofu when compared among samples containing equivalent moisture levels, and (b) the lipid-free samples showed a constant characteristic temperature, below which the fracture stress displayed a plateau, regardless of the initial moisture content. In contrast, the characteristic temperature decreased with a decrease in moisture content when the full-fat tofu was tested (Watanabe *et al.*, 1995a). These differences were seemingly caused by the feature of the lipid the fracture stress of which continued to increase until it was cooled down to −160°C (Watanabe *et al.*, 1995a).

In the present paper, a food was referred to as "completely frozen" when it was cooled lower than or equal to the characteristic temperature, where its fracture stress displayed a plateau.

When frozen food is analyzed by a simple two-component model consisting of pure water ice and CAS, the compressive fracture stress of CAS may be calculated by using an equation (Watanabe *et al.*, 1995a):

$$\frac{\sigma_{\text{food}}}{d_{\text{food}}}=\frac{\sigma_{\text{ice}}}{d_{\text{ice}}}\omega_{\text{ice}}+\frac{\sigma_{\text{CAS}}}{d_{\text{CAS}}}\omega_{\text{CAS}} \qquad (1)$$

where σ is the compressive fracture stress, d the density and ω is the weight fraction, respectively.

The fracture stress of CAS of lipid-free tofu calculated using Eq. (1) and the measured values given in Fig. 1, is plotted in Fig. 2. This calculation required the ice content in the frozen tofu, which was estimated using a "phase diagram" given elsewhere (Watanabe *et al.*, 1995a). This means that tofu was assumed to have moisture-free CAS at a "completely frozen" state.

The average value for the fracture stress of CAS of "completely frozen," lipid-free tofu was 310 MPa regardless of the initial moisture content. This value was 72% larger than that of full-fat tofu (Watanabe *et al.*, 1995a). The cause of this discrepancy may be that the CAS of full-fat tofu contained lipid. There may be a possibility that CAS formed in full-fat tofu is a composite which consists of lipid-free CAS and lipid. In this case, the fracture stress of lipid-free CAS in full-fat tofu may be calculated by extending the two-component model to a three-component model as follows:

$$\frac{\sigma_{\text{food}}}{d_{\text{food}}}=\frac{\sigma_{\text{ice}}}{d_{\text{ice}}}\omega_{\text{ice}}+\frac{\sigma_{\text{CAS}}}{d_{\text{CAS}}}\omega_{\text{CAS}}+\frac{\sigma_{\text{lipid}}}{d_{\text{lipid}}}\omega_{\text{lipid}} \qquad (2)$$

The compressive fracture stress of lipid-free CAS in frozen full-fat tofu was evaluated using Eq. (2) and compressive fracture stress data (reported in the previous paper: Watanabe *et al.*, 1995a) for soybean oil, pure water ice and full-fat tofu. These values are compared in Fig. 3 with that in frozen lipid-free tofu calculated using Eq. (1). A clear agreement between the two was obtained, although the data are scattered due to the brittle nature of the sample. The fracture stress of CAS averaged over the temperatures corresponding to the "completely frozen" state is plotted against the initial moisture content in Fig. 4, which shows the difference between lipid-free CAS and lipid-containing CAS. It is worth noting that the significant scattering of fracture stress shown in Fig. 3 is mainly caused by that of samples with high moisture content. The agreement between the fracture stresses

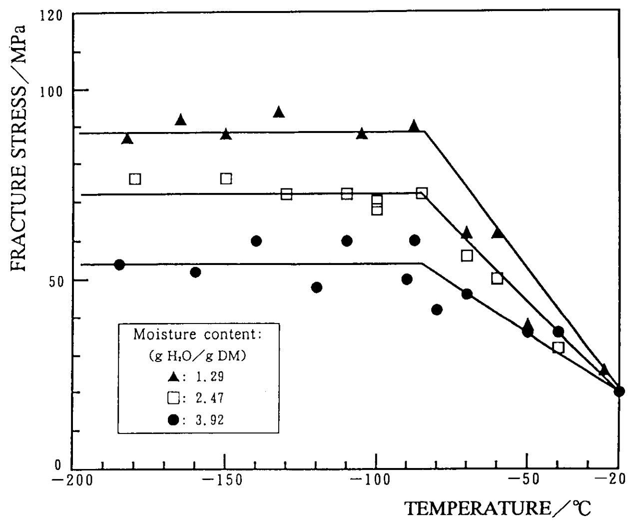

Fig. 1. Compressive fracture stress of lipid-free tofu of selected initial moisture content at temperatures from −20°C to −196°C.

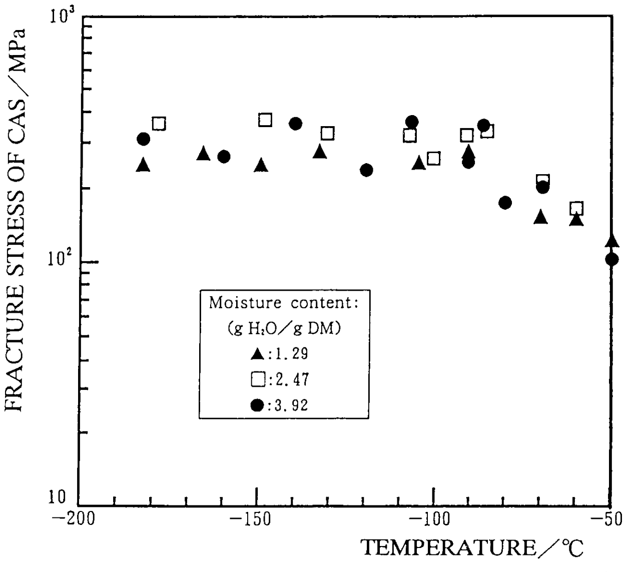

Fig. 2. Fracture stress of CAS of lipid-free tofu calculated using data given in Fig. 1.

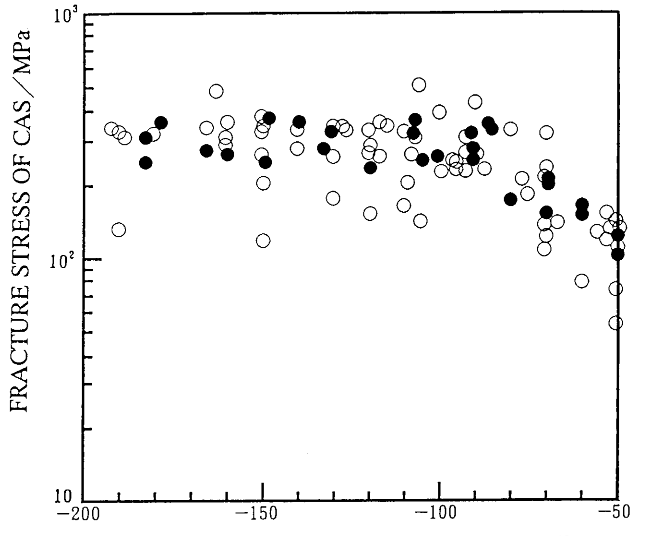

Fig. 3. Fracture stress of lipid-free CAS calculated using Eq. (2) and the data of full-fat tofu (○) and that using Eq. (1) and the data of lipid-free tofu (●).

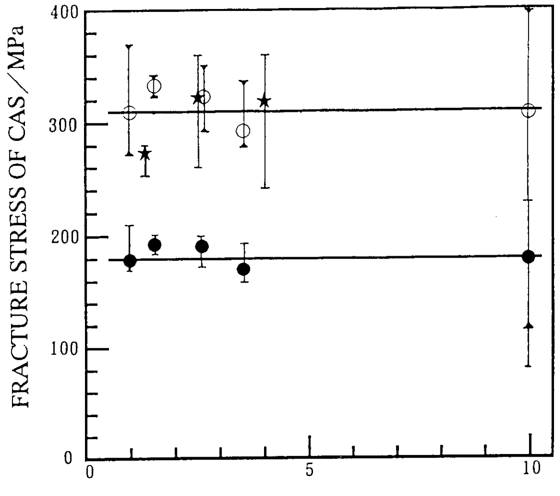

Fig. 4. Compressive fracture stress of CAS averaged over the temperatures corresponding to a "completely frozen" state vs. initial moisture content. Lipid-free CAS in lipid-free tofu (★), lipid-free CAS in lipid-containing tofu (○), lipid-containing CAS (●).

of the two lipid-free CASs supports Eq. (2). This means that the compressive fracture stress of a food is given as the volume-fraction-weighted average of the fracture stress of each component of which the food is composed.

Compressive fracture stress data (the plateau value at "completely frozen" state) of foods other than tofu (the plateau value at the "completely frozen" state) measured in this study as well as those available for comparison (under the same type of "ice capping"), are plotted against the initial moisture content in Fig. 5, which includes the data for dried, smoked bonito meat (containing 2–3% lipid and less than 20% moisture) as well as that for cooked bonito meat (Tang, 1995; Watanabe *et al.*, 1995b). This plot suggests

(a) the compressive fracture stress of lipid-containing foods may be put on a line and those of lipid-free foods on another line, although the value extrapolated to 0% moisture is not clear,

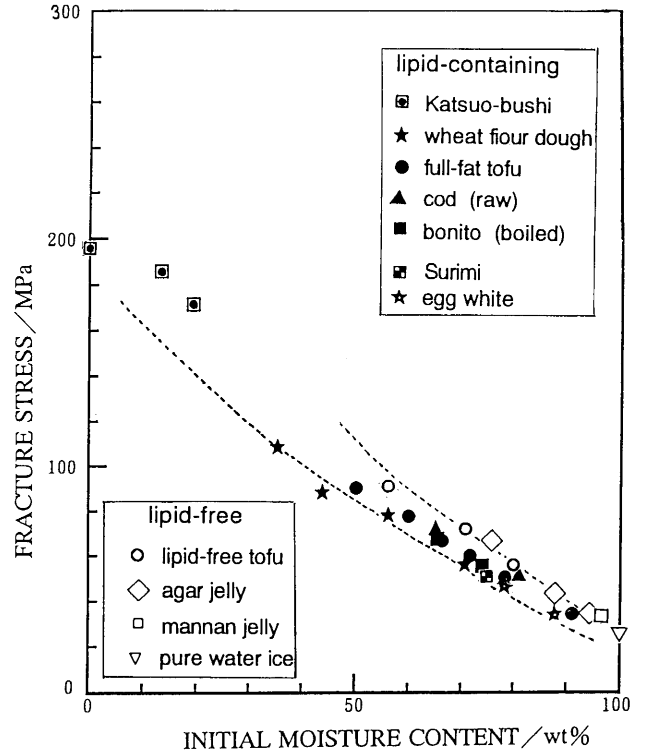

Fig. 5. Averaged fracture stress of selected foods at a "completely frozen" state plotted against initial moisture content.

(b) the fracture stress of completely frozen food is governed by the fraction of ice but affected little by the kinds of biopolymers.

Acknowledgments The authors express their thanks to Ajinomoto Co., Ltd. for offering defatted soy flour.

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