Solidification in Heat Packs: II. Role of Cavitation

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When the metallic trigger in a commercial heat pack is flexed to initiate solidification, the acceleration of the liquid near the solid surface can cause cavitation and collapse of small vapor bubbles. It is investigated whether such cavitation is responsible for the operation of the metallic trigger in the subcooled sodium acetate solution in a heat pack. Using cavity collapse theory and the results in Part I for the nucleation pressure of a sodium acetate solution, it is shown that cavitation can generate pressures greater than the nucleation pressure of the solution. However, the period of time during which the solution is exposed to such high pressure is not sufficient to lead to nucleation, and hence the metallic trigger does not induce solidification as a result of cavitation. Experimental results, using sonication, tribonucleation, and shock waves, confirm the theoretical findings.

Introduction

A heat pack consists of a sealed, flexible PVC envelope (typically 20×10 cm^2) containing a subcooled aqueous solution of sodium acetate (Araki et al., 1995; Ulman and Valentin, 1983). A metallic trigger located within the container initiates the solidification of the liquid to release latent heat (Kapralis et al., 1990). In a subcooled solution, there is a distribution of solid clusters of different sizes. Spontaneous solidification of the solution can only occur when there is an appreciable probability that at least one of these clusters will grow beyond a critical size. A cluster of critical size is called an *incipient nuclei*. In Part I of this series of articles, we used classic nucleation theory to determine the pressure and temperature conditions under which the expected number of incipient nuclei in an aqueous solution of sodium acetate becomes appreciable and nucleation occurs spontaneously. In this article, we investigate if such nucleation in a heat pack can be induced by cavitation and collapse of small vapor bubbles generated during the flexing of the metallic trigger.

Cavitation can be brought about by sonication, tribonucleation (the abrasion of surfaces immersed in solution), propulsion of solid objects through the fluid, or by generating shock waves in a column of fluid. The cavities collapse shortly after forming and during such collapse can generate pressures in the order of thousands of atmospheres in the immediately surrounding liquid (Traverna, 1987). If the pressure in the liquid is greater than its nucleation pressure, the solid clusters in solution will begin to grow spontaneously (see Part I). If they grow to a size greater than the critical size before ambient pressure is reestablished, nucleation and solidification of the entire solution will ensue from that point.

In this work, we describe new experiments carried out in an attempt to achieve cavitation and nucleation in a subcooled solution of sodium acetate. The primary method used to induce cavitation is sonication. Experiments are performed for a range of temperatures. The initial concentration of dissolved gas in the solution, such as CO_2, is also varied. The effect of dissolved gas on the pressures achieved during bubble collapse is difficult to predict (Traverna, 1987). Cavitation is facilitated by a large concentration of dissolved gas, but the speed and severity of collapse, as well as the pressure generated, will be lower. Experiments in this work are performed to both increase and decrease the concentration of dissolved gas in a sodium acetate solution. We also carry out experiments using tribonucleation and the generation of shock waves to induce cavitation in the sodium acetate solution.

We investigate theoretically the mechanism by which cavity collapse may induce nucleation in a subcooled solution. The requirement that the pressure in the liquid is greater than the nucleation pressure is shown to be a necessary but not sufficient condition. In addition, the period of time during which the liquid is subjected to such high pressure must be sufficient to allow the growth of small solid clusters of sodium acetate to the critical size. We calculate these two time scales for nucleation by cavitation in a sodium acetate solution, for a range of temperatures and pressures. The temperature and pressure conditions for which nucleation by cavitation occurs are determined. The theoretical predictions are compared with our experimental results.

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Experimental Procedure and Results

Sonication experiments

Sonication of small volumes ($\sim$25 mL) of sodium acetate solution was carried out using a 20-kHz sonicator capable of producing 475 W. The sonicator tip was thoroughly cleaned before each experiment and inserted into a 50-mL beaker containing the solution. The power delivered by the sonicator was slowly varied from zero to maximum during each experiment in order to test the solution over a wide range of powers.

Experiments were carried out at a standard concentration of $x = 0.206$ mole fraction of sodium acetate in water. The two parameters varied in the experiments were the temperature and the concentration of dissolved gas. The temperature was varied from 273 K to 293 K. At temperatures below about 273 K, it was not possible to assess if cavitation did or did not occur because of condensation on the outside of the beaker. The sonicator was used for only a few minutes in each experiment, so that the temperature of the solution did not rise by more than 5 K. The concentration of dissolved gas in solution was varied by sparging for 30 min with either nitrogen (to strip dissolved gas out of solution) or with carbon dioxide (to saturate the solution with dissolved CO_2).

In each of the experiments just mentioned, the solution became visibly cloudy under the sonicator tip, indicating cavitation. However, nucleation did not occur in any case.

Tribonucleation experiments

Travena (1987) notes that cavitation is thought to be the mechanism responsible for the success of the well-known method of scratching a glass rod against the bottom of a glass beaker to initiate nucleation of organic compounds from solution. Cavitation is thought to occur in the wake of the rapidly accelerating rod as it sticks and slips across the bottom of the beaker.

Glass rods, and stainless-steel spatulas were scratched against the bottom of a glass beaker containing a standard sodium acetate solution, at 273 K. The experiment was repeated after the solution had been sparged with CO_2 and N_2. It is not known whether cavitation occurred because the few cavities expected to be formed by this procedure could not be seen with the naked eye. Nucleation did not occur.

Shock-wave experiments

A small volume of sodium acetate solution of standard concentration, at 294 K, was placed in a glass syringe that was sealed at one end. Drawing back the plunger caused a partial vacuum to be created in the body of the syringe and many cavities appeared in the solution. Releasing the plunger caused it to spring back and collapse the cavities violently. Nucleation, however, did not occur.

The experiments just described represent a good proportion of the experimental methods proposed in the literature to cause the violent collapse of cavities in solution (see Travena, 1987). There are other experiments that allow the processes of formation and collapse of a cavity to be separated and controlled (Hunt and Jackson, 1966). However, these require specialized equipment and were not attempted.

The absence of nucleation and solidification in our experiments is explained in the next sections with reference to the theory of cavity collapse.

Theory

Nucleation can be brought about by the collapse of cavities, because the liquid surrounding the cavity is subjected to extremely high pressures. If this pressure is higher than the nucleation pressure, the solid clusters in solution will begin to grow spontaneously. Solidification of the entire solution will only take place, however, if the period of time during which the solution is subjected to the high pressure is long enough for the growing clusters to reach a size greater than the critical size when the liquid returns to ambient pressure. We therefore need to determine three quantities in order to investigate if the collapse of a cavity can lead to nucleation in a sodium acetate solution. These are the nucleation pressure, the period of time during which the liquid is above the nucleation pressure (the *available time*), and the time necessary for a cluster to grow to a size greater than the critical size (the *necessary time*). Nucleation will occur when the available time, t_a, is greater than the necessary time, t_n. In the next section, we introduce the general theory for growth and collapse of a cavity and calculate the three quantities just given, for a solution of sodium acetate at standard temperature ($T = 294$ K) and pressure ($p = 1$ bar).

Pressure and temperature during cavity collapse

A cavity forms when one portion of liquid is accelerated so rapidly from a neighboring portion of liquid that the bonds between the molecules are broken and a void opens up. The acceleration of the molecules can be brought about by a fast-moving solid surface passing through the liquid or a sonic wave (Travena, 1987). The void will grow for some time, and its maximum size is determined by the liquid properties and the cause of the cavitation. Vapor or gases dissolved in the fluid will evaporate into the cavity as it grows. The bubble starts collapsing when the conditions causing the cavitation change, that is, after the passing of the fast-moving object or of the compression phase of the sonic wave.

The movement of the liquid surrounding the cavity then reverses its direction and the cavity shrinks, compressing the gas inside as it does so. The gas and vapor redissolve and condense as the pressure is increased. The pressure in the liquid immediately surrounding the cavity increases as its momentum is significantly reduced during collapse. The highest pressure is achieved when the cavity reaches its smallest volume, containing gas at very high pressure and temperature. The size of the cavity can then oscillate until the energy of collapse is dissipated as viscous heating, or the shape of the cavity can become unstable during the final stages of collapse and the cavity breaks up violently. For a more detailed general account, the reader is referred to Neppiras (1980) or Kennedy (1998).

Mathematically, the collapse of a cavity is a free boundary type of problem. The governing equations describing the interior of the cavity must be solved simultaneously with those describing the exterior in order to determine the position of the interface with time. The equations describing the interior and exterior of the cavity comprise: (1) conservation of mass for each of the components in the fluid; (2) the equation of motion (conservation of momentum); and (3) conservation of energy. For a detailed description of the formulation of these equations, the reader is referred to Hsieh (1965).

Solving these equations in a general sense is completely impossible. The first assumption usually made is to consider only the collapse of a single isolated cavity that retains its spherical symmetry throughout. Second, the interior of the cavity is assumed to be uniform. The simplification made to the equations by these two assumptions is considerable. The formulation by Gilmore (1952) is used in the present study. The additional assumptions of this formulation are described below in the context of a brief account of the historical development of this problem.

Hsieh (1965) notes that three factors dominate the collapse of a bubble: inertial, thermal, and diffusive. In the case of the collapse of a cavity formed in a subcooled liquid, the inertial effect dominates the other two. Viscous and surface-tension effects are also negligible unless the viscosity or surface tension is unusually high (Ivany and Hammitt, 1965). As a result, the energy equation is decoupled, and the collapse is described by the equations of motion and continuity alone.

During the initial stages of collapse, the speed of the bubble wall and the pressure in the liquid are low. This means that the liquid can be assumed to be incompressible. Rayleigh (1917) considered this case for an empty cavity (but the result is equally valid for the cavity containing only the vapor of the liquid, assuming it remains at its constant equilibrium vapor pressure). He derived an analytic result for the pressure generated and showed that very high pressure could be achieved. Without any insoluble gas in the cavity, the cavity is predicted to collapse completely, and a singularity occurs in the pressure and interface velocity at this point. Rayleigh's solution describes the bubble collapse very well at its initial stages, but an adequate description of the later stages must take into account the fact that liquid compression is significant at such high pressures, and that there will always be some gas remaining in the cavity at its minimum size.

The incorporation of liquid compressibility into the equations was performed by Herring (1941) and Trilling (1952), both of whom assumed a constant speed of sound in the liquid. Gilmore (1952) then allowed for a pressure-dependent velocity of sound. This, however, necessitated a numerical solution to his equations. The incorporation of remaining gas in the cavity into the equations was performed by Hickling and Plesset (1964). Hickling (1965) used these results to show that nucleation of ice in subcooled water by cavitation is possible. We use the same methodology here for a sodium acetate solution.

Sodium acetate solution at standard conditions

The standard conditions for sodium acetate solution are defined as:

(1) Initial temperature of solution: 294 K.

(2) Temperature of the solution surrounding the collapsing cavity: 304 K [it is estimated in Kennedy (1998) that the temperature of the solution surrounding the collapsing cavity increases by about 10 K due to conductive and radiative heating by the hot gases in the cavity].

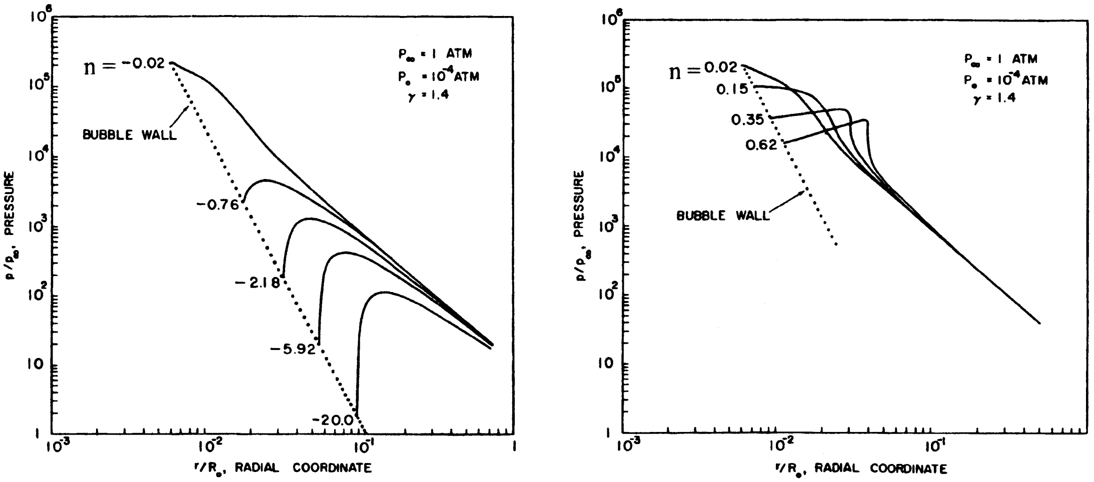
(3) Pressure in the liquid before collapse: 1 bar.

(4) Concentration of solution: $x = 0.206$ mole fraction of sodium acetate in water.

The magnitudes of the nucleation pressure, the time available for nucleus growth, and the time necessary for nucleation are calculated below to illustrate the method of determining whether nucleation is possible by cavitation.

Nucleation Pressure. The relationship between nucleation pressure and temperature was derived in Part I (Eq. 16) as $T_n = 5.1 p_n + 268$, where p is expressed in kbar. The temperature of the fluid surrounding a cavity at standard conditions is 304 K, and therefore the nucleation pressure is 7.1 kbar.

Time Available for Nucleation. The time available for cluster growth is the time the liquid surrounding the collapsing cavity remains above the nucleation pressure of 7.1 kbar. This time can be estimated from the numerical solutions pre-



(*a*) (*b*)

Figure 1. Pressure in the liquid surrounding a cavity during (a) the collapse phase, and (b) the rebound phase.

R_o is the initial cavity size, r is the radial coordinate from the center of the cavity, p is the pressure in the liquid, p_∞ is the initial pressure of the liquid, p_o is the initial pressure of insoluble gas in the cavity, and γ is the ratio of specific heats of this gas. [From Hickling, 1965; reproduced with permission from *Nature*.]

sented in Hickling (1965) for water, assuming the relevant properties of water and sodium acetate solution are similar.

Hickling (1965) considered a cavitation bubble containing insoluble gas at an initial pressure of 10^{-4} bar and having a ratio of specific heats, γ, of 1.4 as typical of a cavitation bubble in water at 1 bar. His results for the variation of pressure in the liquid with distance from the bubble wall are shown in Figure 1. Figure 1a depicts the pressure distribution during the initial collapse phase of the cavity, and Figure 1b shows the pressure during the initial rebound phase of the cavity.

The nondimensional time, n, shown beside each pressure curve is related to dimensional time, t, by the relationship

$$t = n \times \tau / 10^4, \tag{1}$$

where τ is a time scale for collapse, given by Rayleigh's equation

$$\tau = 0.91 R_o (\rho_\infty / p_\infty)^{0.5}. \tag{2}$$

Here ρ_∞ and p_∞ are the initial density and pressure in the liquid. Time, t, is measured from the moment of maximum collapse, that is, $t = 0$ when the bubble size is a minimum. It is negative during collapse and positive after rebound. Figure 1 shows that the pressure at the cavity wall reaches a maximum value of approximately 4×10^5 bar at the moment of minimum cavity size. This peak pressure is the same regardless of initial cavity size, but higher pressure will be maintained for a longer time period when the cavity is larger because of the effect of R_o on the time scale, τ.

The time interval during the collapse and rebound of a cavity for which a particular point on the pressure curve is above the nucleation pressure, of 7.1 kbar, can be determined directly from Figures 1a and 1b. Two points on the pressure curve were chosen for this calculation. The point of peak pressure was chosen as one extreme, leading to an upper bound for the time available for nucleation. The other point we consider is the wall of the cavity, which leads to a lower bound estimate for the time available for nucleation. In Table 1, we show our estimates for the total time, during the collapse phase and rebound phases, during which the liquid at the points described is at a pressure above a given value. A more detailed table showing the interim values obtained directly from Figure 1 can be found in Rogerson (2001).

Table 1. Nondimensional Period of Time, Δn, During Which the Liquid Surrounding a Cavity Is Above a Given Pressure, p_g*

Pressure (p^g/p^∞)	Δn Satisfying Liquid Pressure $p/p_\infty \geq p_g/p_\infty$ At the wall	At peak
2×10^5	0.04	0.04
1×10^5	0.25	0.25
3.5×10^4	0.55	0.75
1.5×10^4	0.9	1.9
2×10^3	3.3	5
2×10^2	10	120
2×10^1	86	600
2×10^0	220	2200

*It includes both the collapse and rebound phases.

Table 2. Crystal Growth Rate of Sodium Acetate Trihydrate from Solution as a Function of Temperature, T, and Mole Fraction of Sodium Acetate, x

Temp., T	$x = 0.206$	$x = 0.170$
273 K	0.44 cm/s	0.23 cm/s
294 K	0.47 cm/s	0.24 cm/s

The results in Table 1 suggest that the time interval during which liquid was subjected to a pressure above 7.1 kbar ($p/p_\infty \geq 7.1 \times 10^3$) is approximately in the range $\Delta n = 2 - 4$. Following the work of Hickling (1965) for water, we assume that the initial size of a cavitation bubble in the sodium acetate solution is typically 10^{-4} m. Then $\tau = 10^{-5}$ s from Eq. 2. Using Eq. 1, we estimate the time available for nucleation, during which a portion of liquid is above a pressure of 7.1 kbar, to be about 3 ns. This available time must now be compared with the time necessary for the growth of a cluster of sodium acetate trihydrate to the critical size.

Time Necessary for Nucleation. The time required for a cluster to grow to a size greater than the critical size at standard conditions of $T = 294$ K and $p = 1$ bar is calculated in this section. The critical size at these conditions was calculated in Part I (Eq. 8) to be 3.5 nm. In order to calculate the time necessary for a cluster to grow to this size, the crystal growth rate needs to be determined.

Four measurements of the growth rate of the solidifying front of sodium acetate trihydrate solution v were made at atmospheric pressure. These measurements were taken by timing the position of solidification of solution in a 10-mL pipette over a total period of time of 20 s. The growth rate was constant for each concentration and temperature. The results are shown in Table 2.

A linear relationship of the growth rate v with temperature and concentration is approximated from these data by

$$v = 9.52 \times 10^{-6} T + 0.061 x - 0.0107 \tag{3}$$

for the range of temperature and concentration investigated.

We now need to consider whether the growth rate is kinetic or diffusion controlled. Diffusion control means that the controlling resistance is the diffusion of excess water away from the growing solid cluster. The growth rate of a highly curved cluster will be greater than the planar growth rate in this case, because the spherical geometry facilitates diffusion. Kinetic control, however, means that the controlling resistance is the rearrangement of molecules at the interface. It is assumed that in this case, the resistance will be similar for the curved and planar cases, and so the linear growth rate measured in Table 2 will be applicable.

Crank (1984) considers a generalized Stefan problem where two media are separated by a moving boundary. The motion of the boundary is determined by diffusion through one of the media. He shows that for diffusion-controlled growth, a planar interface advances as $t^{0.5}$, where t is time. However, our measurements suggest that the growth rate is independent of time, and hence we conclude that the growth of a cluster is kinetically controlled. We shall therefore assume that the planar growth rate given by Eq. 3 describes the growth of a single nucleus and that it is independent of pressure.

At standard conditions of $x = 0.206$ and $T = 304$ K, the crystal growth rate is then 0.0048 m/s. The time necessary for attaining the critical size is calculated by dividing the critical size, r^*, by the rate of growth v giving $t_n \sim 0.73$ μs.

The time required to form a critical cluster, $t_n \sim 0.73$ μs, is over 200 times greater than the time during which the liquid is subjected to sufficiently high pressure, $t_a \sim 3$ ns. This result explains why nucleation did not occur in our laboratory experiments at standard temperature and pressure conditions.

In the next subsection, the effect of changing the temperature, pressure, and concentration of the solution on nucleation by cavitation is investigated.

Nucleation by cavitation at nonstandard temperature, pressure, and concentration

We have seen that at standard temperature and pressure conditions the time available above the nucleation pressure is insufficient to produce nucleation. The theory in Part I suggests that decreasing the temperature or increasing the pressure will favor nucleation. Raising the concentration will also increase the cluster growth rate. There are, in practice, however, limits to the extent to which these parameters can be varied. The solution will become so viscous at low temperatures (~263 K at atmospheric pressure) that violent cavity collapse will be prevented and the concentration can only be raised to the point at which anhydrite will precipitate (see Part I). The pressure, however, can in principle be varied over a very wide range.

As described earlier, the onset of nucleation by cavitation is predicted when the available time at high pressure exceeds the time necessary for nucleation to occur. Using this criterion, the conditions for nucleation of a standard concentration sodium acetate solution have been determined and the results are plotted in $p-T$ space in Figure 2.

Nucleation is predicted to occur in a region bordered by the solid line in the top right of Figure 2. The lower boundary of this envelope, however, is nonphysical, and a consequence of extrapolating the linear variation of C_p with temperature beyond its physical limit, as discussed in Part I. The dashed lines represent a range of physically reasonable extrapolations for the boundary of this region.

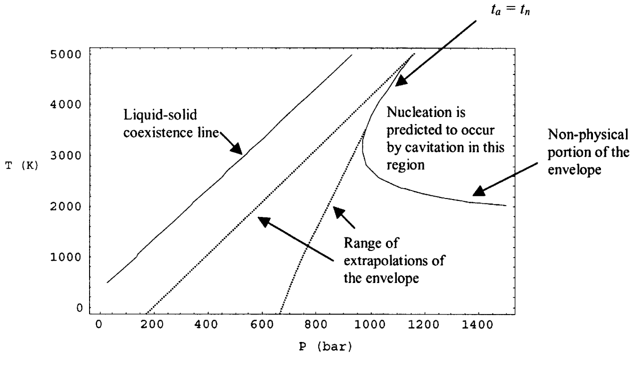


Figure 2. Conditions of temperature and pressure for which nucleation by cavitation occurs in a sodium acetate solution of standard concentration.

It can be seen that even the leftmost extrapolation of the boundaries of this region predicts a minimum initial pressure for nucleation of at least 100 bar at any temperature. The extrapolation is extremely crude, but it shows that formidable pressures are required to cause nucleation by cavitation in sodium acetate trihydrate at any temperature.

Discussion and Conclusions

We have seen that cavity collapse can induce nucleation in a subcooled solution of sodium acetate by rising the pressure in the liquid in the neighborhood of the cavity beyond its nucleation pressure. However, nucleation occurs only if the period of time during which the liquid is subjected to such high pressure is sufficient to allow the growth of solid clusters of sodium acetate to the critical size. We calculated the time available for nucleation during cavitation in a sodium acetate solution at atmospheric pressure to be of the order of 3 ns. The time necessary for cluster growth to the critical size in such a solution was estimated to be 0.73 μs. Comparison of these time scales showed that cavitation cannot induce nucleation in a sodium acetate solution at atmospheric pressure. These theoretical results were supported by our experimental observations that nucleation did not occur in a sodium acetate solution at atmospheric pressure, in which cavitation was promoted by sonication, tribonucleation, or by generating shock waves.

We determined the temperature and pressure conditions for which nucleation by cavitation does occur in a sodium acetate solution. It was found that, at ambient temperatures, an initial pressure in the liquid of at least 200 bar is necessary for nucleation by cavitation. Specialized equipment would be required to undertake experiments at such high pressures, and so experimental evidence to support the theory under these conditions was not sought.

Numerous assumptions have been made in the theoretical analysis, but their effect will not alter the conclusion that extremely high pressures are required for cavitation to be able to induce nucleation in sodium acetate solution. One main assumption is that the typical cavity in a sodium acetate solution has a radius of 10^{-4} m and contains insoluble gas at an initial pressure of 10^{-4} Pa, at all liquid temperatures and pressures. At higher liquid pressures, however, the initial radius of a cavity is expected to be smaller. This would have the effect of reducing the collapse time, and hence reduce the time available for nucleation. Higher initial pressure of the gas in the cavity is also expected at higher liquid pressures; this would reduce the peak pressure reached during collapse. On the other hand, lower initial pressure of gas in the cavity may be achieved by decreasing the temperature of the solution. However, at temperatures much below standard, the solution becomes too viscous for cavity collapse to occur. Therefore our initial assumptions have led to a lower-bound estimate for the pressure required to induce nucleation.

We conclude that cavitation and collapse of any small vapor bubbles generated during the flexing of the metallic trigger in a commercial heat pack cannot be responsible for the triggering of solidification. In Part III of this series of articles, we investigate the hypothesis that the metallic trigger initiates solidification by releasing minute crystals of solid sodium acetate trihydrate into the subcooled solution in a heat pack.

Acknowledgments

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