



# Shock-induced carbonization of phenanthrene at pressures of 7.9–32 GPa

Koichi Mimura<sup>a,\*</sup>, Taisuke Arao<sup>a</sup>, Michiharu Sugiura<sup>b</sup>, Ryuichi Sugisaki<sup>c</sup>

<sup>a</sup>*Division of Earth and Environmental Sciences, Nagoya University, Nagoya 464-8602, Japan*

<sup>b</sup>*Faculty of Pharmacy, Meijo University, Nagoya 468-8503, Japan*

<sup>c</sup>*Department of Teacher Education, Meijo University, Nagoya 468-8502, Japan*

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## Abstract

The chemical behavior of phenanthrene during a reaction triggered by shock waves, and its influence on the physical property of phenanthrene were studied over the pressure range of 7.9–32.0 GPa. Chemical analyses showed that shocked phenanthrene included insoluble carbonaceous material containing amorphous carbon, polycyclic aromatic hydrocarbons (PAHs) with molecular weights ranging from 128 to 356, and unreacted phenanthrene. No diamond and fullerenes were detected in the shocked phenanthrene. The results indicate that reactions triggered by shock wave are dehydrogenation, which causes carbonization and radical addition reactions, and ring cross-linking. Carbonization is the most dominant and rapidly proceeded above 20.0 GPa. Thus, an abrupt increase of compressibility of phenanthrene above 20.1 GPa previously reported is caused by the drastic carbonization.

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## 1. Introduction

When a shock wave is transmitted through a material, the wave produces high pressure–temperature conditions in the material. Under this dynamic condition, the material changes into other materials by shock-induced reactions. The behavior of organic materials under such dynamic conditions has been studied by several researchers [1,2]. It has been reported that several organic materials produce diamond by shock compression [3–5], although the yield of diamond was very low.

Hugoniot plots reflect the physical property of materials under shock compression and have been studied for many materials. Hugoniot plots for various organic materials show some discontinuities in the shock velocity ( $U_s$ )–particle velocity ( $U_p$ ) relation. The discontinuities in  $U_s$ – $U_p$  Hugoniot plots were reported for benzene, anthracene, phenanthrene, and pyrene [1,6]. The discontinuities are thought to be caused by a process producing some

materials with high compressibility different from the starting materials in some pressure range [1,7–9].

The behavior of organic materials has not been described on the basis of the precise chemical analyses of the shocked materials subjected to a wide range of pressures. In this respect, we determined the chemical composition of some products recovered from the shocked sample of phenanthrene at pressures up to 32.0 GPa, and examined the chemical behavior of phenanthrene by the shock reaction and its influence on the physical property.

## 2. Experiments

A schematic cross-section of target assembly for shock–recovery experiment is shown in Fig. 1. The assembly consists of three components: (a) a capsule, (b) a capsule holder, and (c) a momentum trap. The capsule consists of a cap threaded on the inside and a plug threaded on the outside. The thickness of the capsule wall facing the projectile was 1 mm. The plug has a pit 4 mm in diameter and 0.8 mm deep. The assembly was made of stainless steel (SUS 304) and was washed twice with distilled

\*Corresponding author. Fax: +81-52-789-3033.

E-mail address: mimura@eps.nagoya-u.ac.jp (K. Mimura).

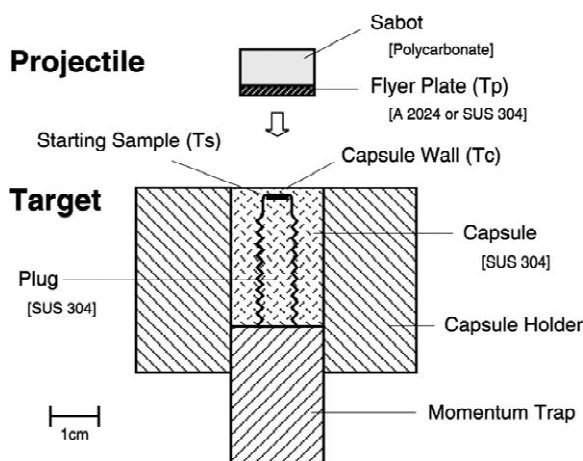


Fig. 1. A cross-section of equipment for shock–recovery experiment.

acetone before use. Phenanthrene was placed into the pit of the plug and pressed to crystal density. The capsule was impacted with a flyer plate, 15 mm in diameter and 2 mm thick, which was glued on a polycarbonate sabot. An aluminum (A 2024) or a stainless steel (SUS 304) flyer plate was used depending upon the desired shock pressure.

In shock–recovery experiments on hydrocarbons, great care must be taken to prevent contamination of the shocked sample. We carefully recovered the shocked sample from the capsule according to the steps reported by Peterson et al. [10]. Recovered samples were split into three parts. One part was analyzed with an elemental

analyzer. Another was analyzed with a fast bombardment mass spectrometer (FABMS) and the other was dissolved in dichloromethane. The solution was filtered to recover and weigh insoluble matter. The recovered insoluble matter was analyzed with an X-ray diffractometer (XRD). The filtrate was concentrated by evaporation and was analyzed by a gas chromatograph with a flame ionization detector (FID-GC), and a GC–MS. For FID-GC and GC–MS analyses, a 30-m $\times$ 0.32-mm fused-silica capillary column coated with a 0.5- $\mu$ m layer of 100% dimethylpolysiloxane was used. The column temperature was programmed from 80 to 325  $^{\circ}$ C at 4  $^{\circ}$ C/min. The temperature for the interface of the GC–MS was 325  $^{\circ}$ C. The concentrations of products in the solution were determined by comparison with those of internal standards.

We estimated the peak shock pressure, the shock temperature, and the shock period (Table 1), on the basis of the equations-of-state for the materials used [11]. The procedure of the estimation is described in Appendix A.

### 3. Results

The elemental composition of shocked samples was hydrogen and carbon, which were determined with an elemental analyzer. Atomic ratios of hydrogen to carbon (H/C) of the samples decrease with increasing shock pressure (Fig. 2). The H/C ratio drastically decreases above 20.0 GPa and falls to 0.07 at 30.5 GPa, while the ratio remains fairly constant below 20.0 GPa. The calculated ratio of lost hydrogen relative to the total hydrogen in phenanthrene as the starting material shows that about 90%

Table 1  
Summary of shock recovery experiments on phenanthrene

Initial density of phenanthrene (g/cm <sup>3</sup> )	Velocity of projectile (m/s)	Flyer plate <sup>a</sup>	Peak shock pressure (GPa)	Shock period ( $\mu$ s)	Shock temp. <sup>b</sup> (K)	Biphenanthrene <sup>c</sup> (354 MW) (%)	Phenanthrene dimer <sup>c</sup> (356 MW) (%)
1.21 $\pm$ 0.01	680 $\pm$ 10	A 2024	7.9 $\pm$ 0.1	0.67	580	n.d. <sup>d</sup>	n.d. <sup>d</sup>
1.21 $\pm$ 0.01	735 $\pm$ 10	A 2024	8.6 $\pm$ 0.1	0.66	600	n.d.	n.d.
1.20 $\pm$ 0.01	970 $\pm$ 10	A 2024	11.7 $\pm$ 0.1	0.64	690	0.01	n.d.
1.20 $\pm$ 0.01	1200 $\pm$ 10	A 2024	14.9 $\pm$ 0.2	0.62	800	0.06	0.02
1.21 $\pm$ 0.01	1410 $\pm$ 10	A 2024	18.0 $\pm$ 0.2	0.60	910	0.35	0.05
1.21 $\pm$ 0.01	1490 $\pm$ 10	A 2024	19.2 $\pm$ 0.2	0.60	960	0.30	0.04
1.21 $\pm$ 0.01	1510 $\pm$ 10	A 2024	19.5 $\pm$ 0.2	0.60	970	0.71	0.06
1.20 $\pm$ 0.01	1550 $\pm$ 10	A 2024	20.0 $\pm$ 0.2	0.59	990	0.78	0.13
1.21 $\pm$ 0.01	1670 $\pm$ 10	A 2024	22.0 $\pm$ 0.2	0.58	1070	1.16	0.24
1.20 $\pm$ 0.01	1760 $\pm$ 10	A 2024	23.4 $\pm$ 0.2	0.58	1130	1.00	0.20
1.20 $\pm$ 0.01	1875 $\pm$ 10	A 2024	25.2 $\pm$ 0.2	0.57	1200	1.24	0.09
1.20 $\pm$ 0.01	1265 $\pm$ 10	SUS 304	27.6 $\pm$ 0.3	0.72	1310	0.40	0.07
1.21 $\pm$ 0.01	1380 $\pm$ 10	SUS 304	30.5 $\pm$ 0.3	0.71	1440	n.d.	n.d.
1.20 $\pm$ 0.01	1435 $\pm$ 10	SUS 304	32.0 $\pm$ 0.3	0.71	1500	n.d.	n.d.

<sup>a</sup> A 2024, aluminum 2024; SUS 304, stainless steel 304.

<sup>b</sup> The shock temperature should be treated as an upper limit.

<sup>c</sup> Concentrations for these compounds are calculated as total value of all identified isomers.

<sup>d</sup> n.d., these compounds could not be detected in shocked phenanthrene.

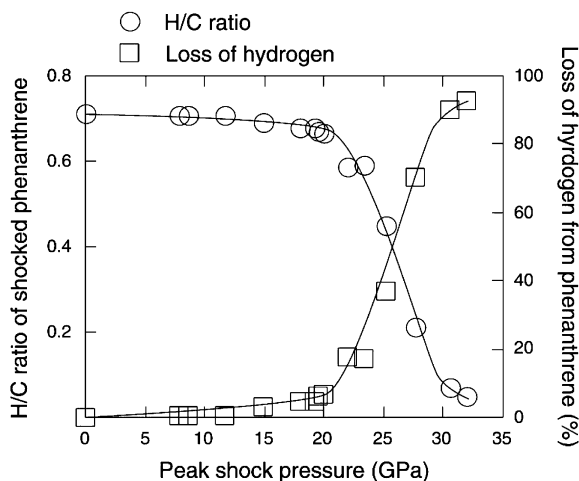


Fig. 2. Atomic ratio of hydrogen to carbon (H/C) of shocked phenanthrene and lost hydrogen from phenanthrene versus peak shock pressure.

of the hydrogen in phenanthrene was degassed during shock compression above 30.5 GPa (Fig. 2). Experimentally, however, hydrogen gas was not determined, because the gas escaped from the shocked sample when the capsule was mechanically cut open to take the sample out.

For all shocked samples, peaks around 720 and 840 atomic mass units were not detected by FAB-MS. This shows that  $C_{60}$  and  $C_{70}$  fullerenes were not produced by shock reaction of phenanthrene.

All shocked samples contained a filtered black matter insoluble in dichloromethane. The concentration of the insoluble carbonaceous material is plotted against shock pressure (Fig. 3). In X-ray diffractograms using  $Cu K\alpha$  radiation the insoluble carbonaceous materials above 14.9 GPa showed a diffuse peak around  $26^\circ$  ( $2\theta$ ), which is practically identical to that of amorphous carbon. Hence, the insoluble carbonaceous material in the shocked sample contains amorphous carbon. The amorphous carbon may include diamond-like carbon (DLC), which consists of a mixture with tetragonal ( $sp^3$ ) and trigonal ( $sp^2$ ) carbon-carbon bonds, although XRD analysis with wider range of  $2\theta$  and Raman analysis were not carried out to identify DLC. The concentration of the insoluble carbonaceous material for samples at 7.9, 8.6, and 11.7 GPa were too low to determine amorphous carbon with XRD. Sharp peaks in the X-ray diffractogram characterizing well-ordered graphite and diamond did not appear in all the shocked samples.

Analyses of FID-GC and GC-MS showed that shocked samples contain many species of organic materials such as polycyclic aromatic hydrocarbons (PAHs) with molecular weights ranging from 128 (naphthalene) to 356 (phenanthrene dimer), including phenanthrene (unreacted phenanthrene). Biphenanthrene and phenanthrene dimer were

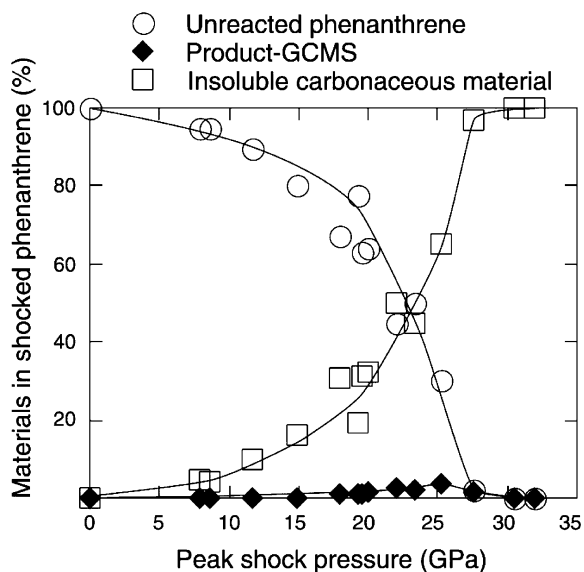


Fig. 3. Concentrations of unreacted phenanthrene, insoluble carbonaceous material, and product GC-MS in shocked phenanthrene versus peak shock pressure.

major products of the shock reaction of phenanthrene and had some structural isomers. Although organic materials with molecular weights lower than 128 and higher than 356 were probably present, they cannot be detected by the present method. Except the unreacted phenanthrene, we designated the PAHs determined by FID-GC and GC-MS as 'product-GCMS.' The unreacted phenanthrene and the product-GCMS are plotted against shock pressure (Fig. 3).

## 4. Discussion

### 4.1. Dehydrogenation

The pressure-dependent H/C ratio of the shocked sample indicates that dehydrogenation characterizes the shock reaction and accelerates rapidly with increasing shock pressure (Fig. 2). This feature correlates with those of the shock reaction for benzene and hexane [12–14]. Mimura [13] and Mimura et al. [14] determined hydrogen gas produced by shock waves for benzene and hexane in closed system experiments, respectively. Ree [9] suggested that hydrocarbons, including phenanthrene, produce hydrogen by dissociation during the shock compression. Nellis et al. [15] thermodynamically predicted the chemical compositions of shocked organics and emphasized hydrogen as one of the major products.

#### 4.1.1. Carbonization

The insoluble carbonaceous material in the shocked samples is probably produced by carbonization, which

produces carbonaceous materials lacking in hydrogen [16,17]. The insoluble carbonaceous material was produced in large amounts above 20.0 GPa (Fig. 3). Since the slope of concentration of the insoluble carbonaceous material for 30.5–32.0 GPa becomes less steep in comparison with that in 20.0–30.5 GPa, the carbonization may be nearly complete around 30 GPa. At 30.5 GPa, the concentration of the insoluble carbonaceous material exceeds 99%. The concentration change of the insoluble carbonaceous material is similar to that of lost hydrogen from phenanthrene (Figs. 2 and 3). Thus, carbonization is the most important mechanism in the shock reaction and strongly affects the behavior of phenanthrene during shock.

The pressure and temperature conditions of the present study are plotted in the phase diagram of carbon according to Bundy et al. [18] (Fig. 4). As this figure shows, diamond is expected to be produced in our experiments. However, in all the shocked samples no diamond was detected. There are two possible explanations. The first is that the chemical structure of phenanthrene containing condensed benzene rings is responsible for the diamond deficiency. Wentorf [19] exposed various organic materials to high pressures (9.5–15 GPa) and high temperatures (1300–3000 °C) for periods of 0.2–50 min. He found diamond production for some specific organics, but did not detect diamond from the condensed-ring compounds, where he found graphite. He concluded that the condensed-ring compounds such as naphthalene, anthracene, and chrysene pyrolyze to graphite and are unusually reluctant to form diamond. Wentorf's results correspond with ours regarding the diamond deficiency. However, we did not detect graphite but amorphous carbon in our shocked samples. The essential difference between the two experiments is the reaction period. The reaction period in the shock experiments may be too short to crystallize carbon

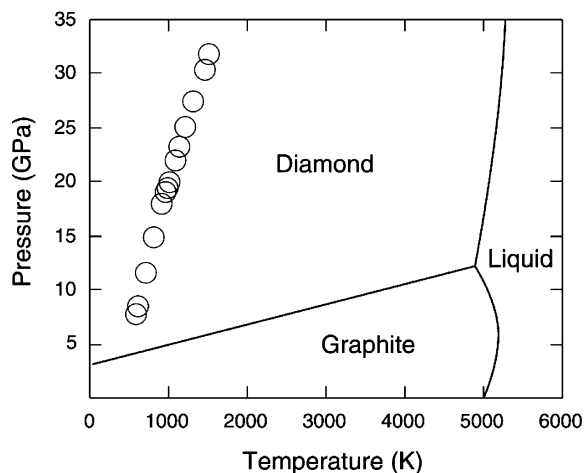


Fig. 4. Carbon phase diagram according to Bundy et al. [18]. Open circles show our calculated data.

as graphite (Table 1). The second explanation is that post-shock heat altered or oxidized the diamond that might have been synthesized. The post-shock heat is generated by an increase of internal energy that is caused by the difference between shock compression (irreversible adiabatic process) and pressure release (isentropic process). The temperature controlled by the post-shock heat is somewhat lower (e.g., approximately 610 K at 32 GPa) than the shock temperature and decreases to ambient temperature within an order of 10 ms by thermal diffusion. Therefore, post-shock heat does not seem to be responsible for the absence of diamond.

#### 4.1.2. Radical addition reaction

Biphenanthrene with molecular weight (MW) of 354 was the dominant component in the product GC–MS. The formation of biphenanthrene was prominent in the pressure range of 18.0–25.2 GPa, and its maximum value (1.24%) appeared at 25.2 GPa (Table 1). Biphenanthrene is formed from two molecules of phenanthrene (178 MW) by eliminating one hydrogen molecule. One possible mechanism for the formation of biphenanthrene is a radical addition reaction. In this reaction, a cleavage of carbon–hydrogen bond occurs to produce highly reactive radicals such as a phenanthryl radical, which polymerize and form biphenanthrene. This reaction tends to occur at high temperatures. Lewis [16] converted naphthalene (128 MW) and anthracene (178 MW) to pitch-like products by heat treatment in closed vessels at 500 °C for 50 h and detected mixtures of polymers including binaphthalene (254 MW) and bianthracene (354 MW), respectively. He attributed the polymer formation to the radical addition reaction.

The other possible mechanism is ring-cross linking (detailed in the next section) followed by the elimination of hydrogen. In the first step of this process, the ring-cross linking of two phenanthrene molecules occurs and produces phenanthrene dimer (356 MW). In the second step, a cleavage of one  $\sigma$  bond in the phenanthrene dimer with dehydrogenation takes place and subsequently biphenanthrene forms.

#### 4.2. Ring cross-linking

Phenanthrene dimer (356 MW) was one of the major products (Table 1). Phenanthrene dimer should be a face to face doubly cross-bonded compound, because there is no mass loss in its formation. The possible mechanism for formation of phenanthrene dimer is ring cross-linking in which intraring  $\pi$  bonds are converted to interring  $\sigma$  bonds. It has been accepted in the field of physical chemistry that the formation of dimers results in the compressibility change of materials under shock compression, but the dimers in the shocked materials could not be identified. Ring cross-linking is known to be driven by pressure [20–22]. Warnes [1] expected that ring cross-

linking occurs in the shock reaction of anthracene, but he could not detect anthracene dimer (356 MW) as a product of the cross-linking. For this result, he suggested that the chaotic conditions after shock might have changed an initially cross-linked material such as anthracene dimer to other materials. Engelke and Blais [7] detected materials with molecular weights ranging from 350 to 360 in shocked anthracene and concluded that these materials were produced by the pressure-driven ring cross-linking.

#### 4.3. Influence of carbonization on compressibility

Phenanthrene was changed to other materials by shock reactions, especially carbonization. Carbonization should influence the physical property of phenanthrene under shock compression. Fig. 3 shows that unreacted phenanthrene was dominant in the shocked samples up to 20.0 GPa; the concentration of the unreacted phenanthrene was comparable with that of insoluble carbonaceous material at 22.0 GPa. The insoluble carbonaceous material was dominant above 25.2 GPa and reached nearly 100% at 32.0 GPa. In the pressure range between 7.9 and 20.0 GPa, phenanthrene was still the dominant component and carbonization hardly occurred. Thus, physical properties in this range will maintain the features expected from that of phenanthrene. The property in the range of 20.0–32.0 GPa must show a different mode from that in the range 7.9–20.0 GPa, because carbonization rapidly proceeds above 20.0 GPa and produces a large amount of insoluble carbonaceous material. Dick [8] also tried to interpret changes of compressibility indicated by the Hugoniot for phenanthrene on the basis of the composition of shocked phenanthrene, although the interpretation was not based on experimental data. He separated the Hugoniot for phenanthrene into three segments and designated the pressure ranges below 20.1 GPa, between 20.1 and 32.5 GPa, and above 32.5 GPa as regions I, II, and III, respectively (Fig. 5). He suggested that ‘transition’ of phenanthrene starts at 20.1 GPa and is completed at 32.5 GPa, and that the shocked phenanthrene between 20.1 and 32.5 GPa is in a mixed phase, and argued that the transition is responsible for the distinction between regions I, II, and III.

The reason why the shocked phenanthrene changes its compressibility in some range of shock pressures can be provided by the chemical composition of the shocked sample as determined in our experiments. In shocked anthracene above the pressure (17.6 GPa) of the first cusp in the  $U_s-U_p$  Hugoniot, Warnes [1] and Engelke and Blais [7] detected an organic material with 354 MW and organic materials with 350–360 MW, respectively. On the basis of these results, they concluded that the increase in compressibility above 17.6 GPa should be associated with the dimerization of anthracene. If it is possible to apply this conclusion to discussion on shocked phenanthrene, the increase in compressibility between 20.1 and 32.5 GPa should be due to the formation of biphenanthrene and

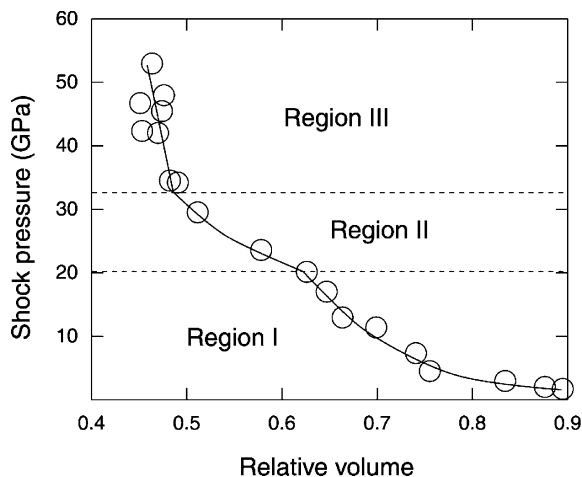


Fig. 5. Relationship between relative volume of shocked sample and shock pressure, which was plotted using Warnes's data [1]. Regions I, II, and III were designated by Dick [8].

phenanthrene dimer. In our experiment, these products were detected in the shocked phenanthrene. However, the concentration of these products is less than 1.4%. Therefore, the increase in compressibility between 20.1 and 32.5 GPa cannot entirely be attributed to the formation of biphenanthrene and phenanthrene dimer. On the other hand, Ree [9] suggested that hydrocarbons containing phenanthrene dissociate into two separate condensed phases of carbon and hydrogen under shock compression; the former represents diamond and the latter a molecular fluid hydrogen or a solid hydrogen. He concluded that the discontinuous change in compressibility of the hydrocarbons with pressure must be attributed essentially to the volume change of the molecular hydrogen, because the specific volume of diamond is relatively insensitive to pressure. In our study, the amounts of insoluble carbonaceous material and hydrogen increased drastically above 20.0 GPa, and diamond was not detected. Therefore, the formation of the insoluble carbonaceous material as well as hydrogen should be responsible for the high compressibility. No compositional data for shocked phenanthrene above 32.0 GPa were available in the present study. However, a relatively small compressibility above 32.5 GPa previously reported [1] may be attributed to the insoluble carbonaceous material containing some material (e.g., diamond), which hardly changes its specific volume with pressure.

## 5. Conclusion

The shocked samples of phenanthrene contained insoluble carbonaceous material containing amorphous carbon and PAHs with molecular weights ranging from 128 to 356, as well as unreacted phenanthrene. The shock reaction can be essentially regarded as carbonization. The carboni-



zation drastically increased above 20.0 GPa and produced an insoluble carbonaceous material. An abrupt increase in the compressibility of phenanthrene above 20.1 GPa is chiefly attributed to the drastically compositional change caused by the production of the insoluble carbonaceous material and hydrogen. The formation of biphenanthrene and phenanthrene dimer does not affect the compressibility, although their formation should play an important role in the shock reaction.

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### Appendix A

When the thickness of the flyer plate ( $T_p$ ), the capsule ( $T_c$ ), and the sample ( $T_s$ ) satisfy the conditions of  $T_p > T_c + T_s$  and  $T_c > T_s$  (Fig. 1), one can assume that a planar shock wave traverses the relatively thin sample and that the sample equilibrates with the pressure of the surrounding metal by repeated reflections of shock wave within the capsule. Thus, on the basis of the equations-of-state of the metals used [11], the peak shock pressure in the sample can be determined from the projectile velocity, which was measured by the flight time between two laser beams. The sample in the capsule is not uniformly shocked to the same conditions, because a radial pressure release at the edge of the sample occurs. Therefore, a part of the sample would be subjected to lower pressure than the calculated peak shock pressure. This edge effect forces us to underestimate the effect of the shock pressure on the shock reaction of phenanthrene. The edge effect should be examined for various sample thicknesses.

The shock period was estimated to be the round-trip time of the shock wave in the flyer plate. The initial shock wave traveling rearward in the flyer plate is reflected at the free rear surface as a tensile rarefaction wave. When this rarefaction wave enters the target, the sample will be decompressed.

The shock temperature was calculated on the basis of Marsh's data [11] and the following equation

$$Cv = ((\partial E / \partial V)_H + P) / ((\partial T / \partial V)_H + \gamma T / V),$$

where  $P$ ,  $V$ ,  $E$ , and  $T$  are the Hugoniot pressure, volume, internal energy, and temperature, the partial derivatives are taken along the Hugoniot, and  $\gamma$  is the Grüneisen  $\gamma$ . In this

calculation, the Grüneisen  $\gamma$  and specific heat constant were taken as approximately  $2S - 1$  and  $3R$  per mol atom, respectively.  $S$  and  $R$  show the bulk modulus and the gas constant, respectively. The shock temperature is influenced by shock-wave structure [23]. For the same terminal shock pressure, a single shock compression produces a higher shock temperature than a multiple shock compression. Although phenanthrene samples experienced multiple shock compressions in the present experiment, we calculated the shock temperature based on a single shock. Accordingly the calculated result involved some error and the calculated shock temperatures should be treated as upper bounds.

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