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Time Response of Anodized Aluminum Pressure-Sensitive Paint

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The response time of anodized aluminum pressure-sensitive paint (AA-PSP) was studied to develop the capability of making global pressure measurements in unsteady flow conditions. This pressure-sensitive paint (PSP) uses anodized aluminum as a porous supporting matrix, which improves the response time by increasing the oxygen diffusion process in the PSP layer. A response time of 34.8 μs was obtained from AA-PSP with 4.3-μm matrix thickness. Response times of PSPs were measured from a step change of pressure created by a normal shock wave in a shock tube. The response time of AA-PSP is dependent on the matrix thickness, with a thin supporting matrix giving a fast response time. Compared with other porous PSPs, such as thin-layer chromatography PSP, porous polymer/ceramic PSP, and conventional polymer matrix PSP, AA-PSP has the fastest response time. The material of the supporting matrix greatly changed the response time of the PSP. The increase in surface temperature of the paint caused by shock waves was also measured using a temperature-sensitive paint (rhodamine-B on anodized aluminum) to understand the temperature effect of AA-PSP.

Nomenclature

A, B	=	calibration coefficients in Eq. (6)
b_F	=	Freundlich isotherm coefficient
D	=	mass diffusivity of oxygen
d_{spot}	=	laser spot size
I	=	luminescent intensity
K_q	=	Stern–Volmer constant
l	=	thickness of a supporting matrix
$[O_2]$	=	oxygen concentration
$[O_2]_{\text{ads}}$	=	oxygen concentration on a porous surface
$[O_2]_{\text{ads }M}$	=	maximum oxygen concentration on a porous surface
p	=	static pressure
p_1	=	driven pressure
p_2	=	static pressure after a normal shock wave
p_4	=	driver pressure
p_5	=	static pressure after a reflected shock wave
t_{limit}	=	limitation of a pressure rise time
γ	=	Freundlich isotherm parameter
Δd	=	distance between the pressure transducer and a laser spot on the pressure-sensitive paint (PSP)
Δt	=	time delay between the pressure transducer and a laser spot on the PSP
θ	=	fractional coverage of oxygen on a porous surface
v_s	=	speed of a shock wave
τ	=	response time

Subscripts

ref	=	reference condition
0	=	without quencher

Introduction

PRESSURE-SENSITIVE paint (PSP) measures surface pressure distributions based on photoluminescent phenomena.[1] The advantage of this technique over conventional pressure tap and pressure transducer measurements is the ability to make global surface pressure measurements at a lower cost. In the presence of oxygen in a test gas, the luminescent intensity is reduced by oxygen quenching. Because the amount of oxygen in the test gas can be related to static pressures, one can obtain pressure signals from the luminescent intensity of PSP.

PSP consists of two components: luminescent molecules (luminophore) and a supporting matrix. Usually, a polymer is used as the matrix. The luminophore is mixed in this matrix layer, and the resultant paint is applied onto wind-tunnel model surfaces, usually by spraying.

PSP has been widely used in steady flow conditions.[2,3] However, surface pressure measurements in unsteady flow conditions have not been well demonstrated. The capability of measuring surface pressure distributions in unsteady flow conditions is limited by the response time of the PSP. Response time is largely dependent on the matrix characteristics, which limit the oxygen diffusion process through the PSP layer.[4]

In this paper, fast response time anodized aluminum PSP (AA-PSP) is presented. Anodized aluminum is a porous material, which improves the oxygen diffusion process in a PSP layer. The AA-PSP demonstrated a fast response time when tested in a shock tube, in which a normal shock wave creates a pressure jump on the order of a few microseconds. The temperature and the thickness dependencies of the supporting matrix for the response time of this PSP are considered, and comparisons to porous and conventional PSPs are made.

Background

Anodized Aluminum PSP

Because of its porosity, a porous supporting matrix of anodized aluminum has a large surface area. Luminophores are applied to this porous surface. Unlike polymer matrices used in conventional PSPs, oxygen molecules, diffusing inside pores, directly interact with luminophores by gaseous collision and adsorption to cause oxygen quenching. Figure 1 shows a comparison between AA-PSP and conventional PSP. A porous structure enables AA-PSP to have a high mass diffusivity of oxygen through the PSP layer. The anodized aluminum was first applied to PSP by the Central Aero-Hydrodynamic Institute (TsAGI).[5] Asai et al. used this technique for pressure measurements in a cryogenic wind tunnel.[6] Sakaue et al. improved the technique to reduce aging and to make uniform pressure sensitivity on the surface.[7] The stability of AA-PSP has been tested for a time period of over four months in a wind tunnel. The test showed the pressure sensitivity decreased by tens of percent.

Figure 2a shows a scanning electron microscope (SEM) picture, and Fig. 2b shows a schematic of an anodized aluminum surface. Micropores of 20–100 nm in diameter are distributed on the surface. The thickness of anodized aluminum is increased by increased anodization time. The actual increase of model thickness by anodization is on the order of a few micrometers because the supporting

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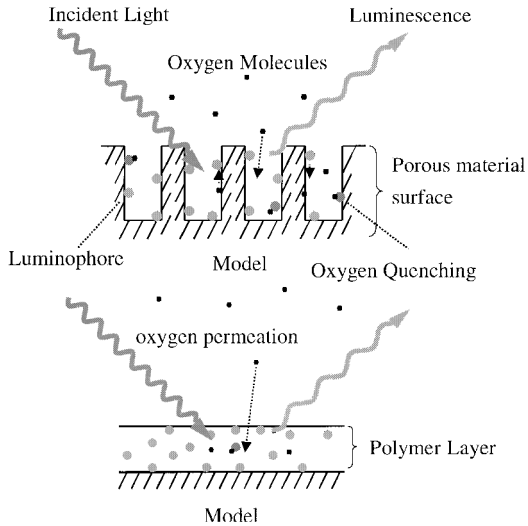
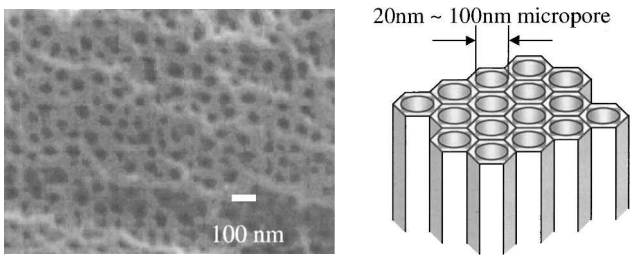


Fig. 1 Comparison of AA-PSP (porous PSP) and polymer PSP (conventional PSP).



a) SEM picture **b) Schematic**

Fig. 2 SEM picture and schematic of an anodized aluminum binder surface.

matrix grows inward and outward to the model surface. The matrix is formed on an aluminum wind-tunnel model by the electrochemical process of anodization. When this process is used, robust anodized aluminum matrices can be formed on complex surfaces. Other candidate substrates that create porous structures may be titanium and stainless steel. However, further investigations of these materials are necessary.

Adsorption Controlled Model

The relationship between the luminescent intensity and surface pressures of a wind-tunnel model can be derived from the Stern–Volmer equation,

$$I_0/I = 1 + K_q[O_2] \tag{1}$$

where I_0 is the luminescent intensity without quencher and I is the luminescent intensity with quencher. The quencher is oxygen, which is described by the oxygen concentration $[O_2]$.

The oxygen quenching process for porous PSP is controlled by adsorption and surface diffusion of the adsorbed oxygen on a porous surface. The oxygen concentration on a porous surface $[O_2]_{ads}$ can be described by the fractional coverage of oxygen θ:

$$\theta = [O_2]_{ads}/[O_2]_{ads\,M} \tag{2}$$

where $[O_2]_{ads\,M}$ is the maximum oxygen concentration on a porous surface. Carraway et al. proposed the oxygen quenching mechanisms of ruthenium complexes on porous fumed silica[8] based on langmuir and Freundlich isotherms. The difference between the two isotherms is that the langmuir isotherm assumes an ideal adsorption that holds uniform interaction energy between the oxygen and pores up to monolayer coverage and the Freundlich isotherm is an extension of the langmuir isotherm that provides an empirical parameter for variation in the interaction energy. From their investigation,[8] the best fit of the quenching mechanism was the model derived from the Freundlich isotherm. The fractional coverage θ can be related to the static pressure p by the Freundlich isotherm,

$$\theta = b_F p^{\gamma} \tag{3}$$

where b_F and γ are functions of temperature. When Eqs. (2) and (3) are substituted into Eq. (1),

$$I_0/I = 1 + K_q[O_2]_{ads\,M} b_F p^{\gamma} \tag{4}$$

Usually, it is difficult to obtain a zero oxygen condition for aerodynamic applications. One can set a reference condition, such as

$$I_0/I_{ref} = 1 + K_{q\,ref}[O_2]_{ads\,M} b_{F\,ref} p^{\gamma\,ref} \tag{5}$$

Dividing Eq. (4) with a reference condition (5), one can derive the adsorption controlled model (Freundlich type)

$$I_{ref}/I = A + B(p/p_{ref})^{\gamma} \tag{6}$$

where

$$A = 1\Big/\left\{1 + K_{q\,ref}[O_2]_{ads\,M} b_{F\,ref} p_{ref}^{\gamma\,ref}\right\}$$

$$B = \frac{K_q[O_2]_{ads\,M} b_F p_{ref}^{\gamma}}{1 + K_{q\,ref}[O_2]_{ads\,M} b_{F\,ref} p_{ref}^{\gamma\,ref}}$$

Here the subscript ref denotes reference conditions with known pressure and temperature.

PSP Response Time

The response time of conventional PSP is proportional to the matrix thickness squared, l^2, and the inverse of the mass diffusivity of oxygen D in a PSP layer[4,9]:

$$\tau \propto l^2/D \tag{7}$$

Normally, conventional PSP has a response time on the order of fractions of second.

Three approaches have been used to improve the response time of PSP. Two of them are applied to a conventional PSP, which uses a polymer matrix. One of the approaches is to develop a PSP with the luminophore concentrated on the top of a matrix surface. Carroll et al.[4] reported a response time of 45 ms was obtained when they sprayed the luminophore over the top of a white primer with a matrix thickness of 19 μm. Another approach is to reduce the PSP matrix thickness. Hubner et al.[10] used this approach and reported that response times of 3–6 ms were obtained from PSPs with polymethylsiloxane and with epoxy/silica gel as matrices by reducing the matrix thicknesses to between 4 and 5 μm.

The other approach is to develop a PSP with high mass diffusivity by using a matrix material other than a polymer. This was reported by Baron et al.[11] by using a commercial porous silica thin-layer chromatography (TLC) plate as a supporting matrix. Although submillisecond response times were obtained by this PSP, it is fragile and limited to simple shapes. However, the work of Baron et al. suggests that fast response time could be obtained by using porous material as a supporting matrix. Anodized aluminum is used as a PSP matrix to achieve a high mass diffusivity and to reduce the matrix thickness.

Experimental Setup and Procedure

PSP Preparation

From AA-PSP considerations in our previous work,[7] the porous supporting matrix of anodized aluminum was prepared from pure aluminum, and the top surface of the matrix was removed to prevent aging and nonuniform calibration constants. The top surface of anodized aluminum was removed by dipping in phosphoric acid. Bathophen ruthenium was used for pressure measurements, and rhodamine-B was used for temperature measurements. These luminophores were dissolved in dichloromethane, and the anodized

aluminum was dipped into the dichloromethane solution to apply the luminophore.

The thickness of AA-PSP matrix can be controlled by the anodization time.[7] In this paper, the anodization times of 30, 60, 90, and 180 min were chosen. These anodization times correspond to binder thicknesses of 4.3, 9.0, 13.2, and 27.2 μm, respectively, measured by an eddy current probe (Kett; Model LZ-330).

Two other porous materials were used as porous supporting matrices: TLC plate and porous polymer/ceramic (PC).[12] In addition, a conventional PSP with silicone rubber as a matrix was considered. These PSPs were used to compare the results from AA-PSP.

The TLC plate was obtained from Aldrich Chemicals. Bathophen ruthenium was applied to the TLC surface by dipping the TLC plate into a bathophen ruthenium in dichloromethane solution. PC-PSP was obtained from Scroggin.[12] This PSP uses ceramic particles with a small amount of polymer. Platinum tetrakis (pentafluorophenyl) porphyrin was used as a luminophore. The polymer PSP was prepared from the bathophen ruthenium mixed in room temperature vulcanized rubber. The thicknesses of TLC-PSP, PC-PSP, and polymer PSP were 152, ~50, and ~60 μm, respectively.

Shock Tube Setup

The response time of PSPs has been measured from a step change of pressure. A solenoid valve-type switching is normally used to create the step change.[9,13–15] This type of apparatus has a pressure rise time on the order of milliseconds. Another method for creating a step change of pressure is to use a shock tube.[10,16] This apparatus creates a normal shock wave, which travels inside the tube. A discontinuous pressure change across the shock creates a step change of pressure. The pressure rise time of this type of apparatus is on the order of microseconds.

Figure 3 shows a schematic of the shock tube setup. The shock tube has a cross section of 55 by 40 mm rectangular, with a driver section length of 428 mm and a driven section length of 485 mm. Aluminum foil was used as a diaphragm material. The diaphragm was burst by the pressure difference between driver and driven sections, where the driver pressure is 1 atm.

A pressure transducer from PCB Piezotronics model 103A11 (unsteady reference) was used for unsteady measurements. This transducer was mounted on the shock tube wall with a 2-mm-diam pressure tap. Absolute pressures were measured by an Omega pressure transducer, which was connected to the driven section. PSP samples were applied to a 25.4-mm square aluminum block. The aluminum block is flush with the shock tube wall so that pressure variations are not created by the sample.

The unsteady reference transducer and PSP sample were mounted 300 mm from the diaphragm. The distance between the pressure transducer and a laser spot on the PSP, Δd, causes a time delay between the two, Δt:

$$\Delta t = \Delta d / v_s \tag{8}$$

A 1-mm Δd causes ~2 μs of Δt depending on v_s. Even though carefully aligned, a small difference of the transducer and PSP sample causes the time delay. This time delay Δt was taken into account to correct the timescale of PSP and pressure tap measurements.

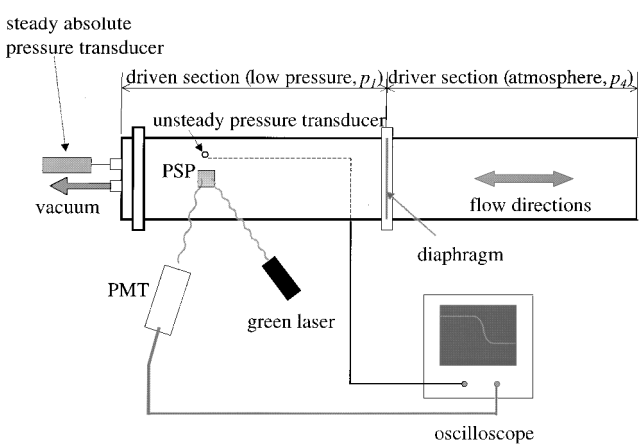


Fig. 3 Schematic of shock tube setup.

A 532-nm laser was used as an illumination source for PSPs with a laser spot size of ~2 mm. The luminescent intensity from the PSP was collected by a photomultiplier tube (PMT) through a 570-nm high-pass filter. The response time of the PMT was 2 μs, measured by the time delay from square waves. Readout voltage from the PMT was collected by a LeCroy oscilloscope at a sampling rate of 1 ms/sample for conventional PSP and 20 ns/sample for the others. The trigger was set at the first step change of PMT voltage, which corresponds to the first step change of the pressure.

A main measurement limitation of the apparatus is laser spot size. A laser spot size d_{spot} with a known shock velocity v_s gives a limitation of a pressure rise time t_{limit} for a PSP:

$$t_{\text{limit}} = d_{\text{spot}} / v_s \tag{9}$$

A smaller spot size photodegrades a PSP, and a larger spot size increases t_{limit}, although a high signal-to-noise ratio is obtained. In our apparatus, t_{limit} is 3–5 μs. PSP signals are low-pass filtered from $1/t_{\text{limit}}$ Hz to reduce the PMT shot noise.

Theoretical calculations were made to predict the first step change of the pressure and the running time of the step.[17] Figure 4 shows a theoretical calculation of the x–t diagram with the initial driven and driver pressures of 25.6 and 100 kPa, respectively. The initial time corresponds to the time when the diaphragm is burst. An incident normal shock wave passes the PSP sample and is reflected at the end wall of the tube. The PSP sample measures the pressures of the initial driven pressure p_1, after the normal shock wave, p_2, and after the reflected normal shock wave, p_5. The magnitude of p_2 and p_5 are dependent on the initial driver and driven pressures, p_1 and p_4, respectively. In our initial conditions, p_2, and p_5 are 48.9 and 88.1 kPa, respectively.

Calibration

The luminescent intensity signals from PSP were converted to pressures through calibration curves. Two calibration methods are available: a priori calibration and in situ calibration. The a priori calibration curve was obtained before the shock tube experiments. The pressure inside the driven section was changed from near vacuum to atmospheric pressure, and corresponding luminescent intensity signals were measured. In situ calibration uses luminescent intensity signals from a PSP and pressure data from the unsteady reference transducer in shock tube experiments. This method improves the accuracy of the pressure measurements. For both calibration methods, the adsorption controlled model in Eq. (6) was applied to determine calibration constants.

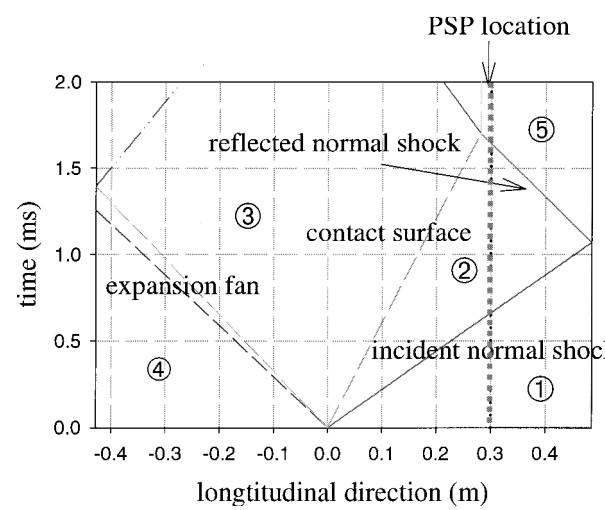


Fig. 4 Calculated x–t diagram of the shock tube; initial driver and driven pressures are 100 and 25.5 kPa, respectively. PSP locates at 300 mm from the diaphragm at x = 0; initial time corresponds to the burst time of a diaphragm.

Results and Discussion

Temperature Dependency

Temperature signals were obtained from a temperature probe (rhodamine-B) applied on anodized aluminum temperature-sensitive paint. The a priori temperature and pressure calibration curves for bathophen ruthenium [Ru(dpp)] are shown in Figs. 5a and 5b, respectively. There was a ~2 K temperature increase on the shock tube wall as the incident normal shock wave passed, as shown in Fig. 6. This temperature increase corresponds to a 0.5% decrease in luminescent intensity. Pressure transducer signals are shown for comparison, and the scale is shown in the right-hand axis. The pressure uncertainty of AA-PSP [Ru(dpp) as a luminophore] due to this temperature change is 0.54 kPa. This pressure uncertainty is determined from a priori calibration curve shown in Fig. 5b with the intensity decrease of 0.5%. A 4.8 kPa change is 2.3% of the pressure change created by an incident normal shock wave.

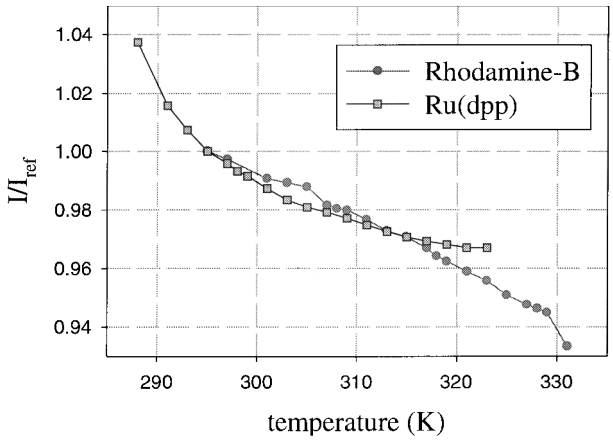


Fig. 5a Temperature calibration curves of rhodamine-B and Ru(dpp) on anodized aluminum.

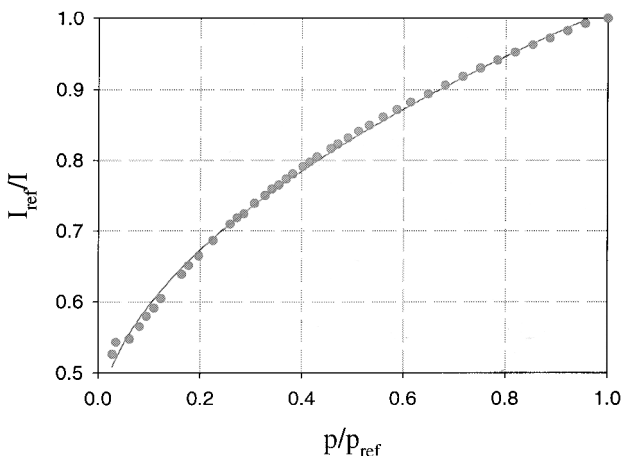


Fig. 5b A priori calibration curves of Ru(dpp) on anodized aluminum (P_{ref} = 101 kPa and T_{ref} = 290 K).

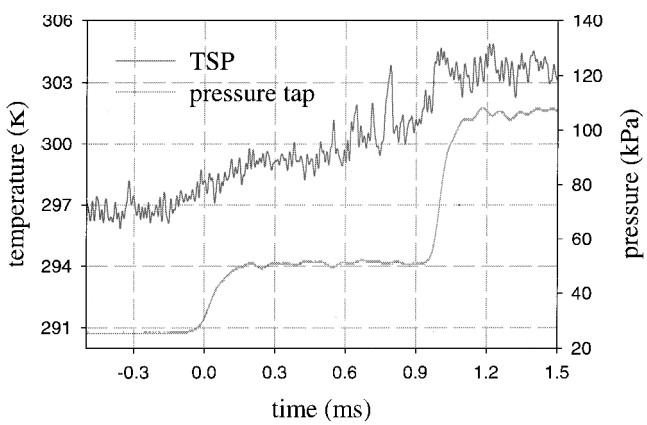


Fig. 6 Temperature and pressure measurements, obtained from TSP (rhodamine-B on anodized aluminum) and pressure tap.

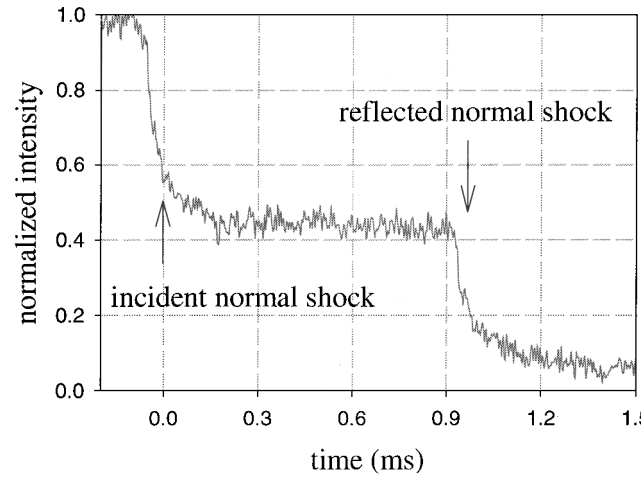


Fig. 7 Luminescent intensity signal of AA-PSP with the supporting matrix thickness of 9 μm shown in a normalized scale.

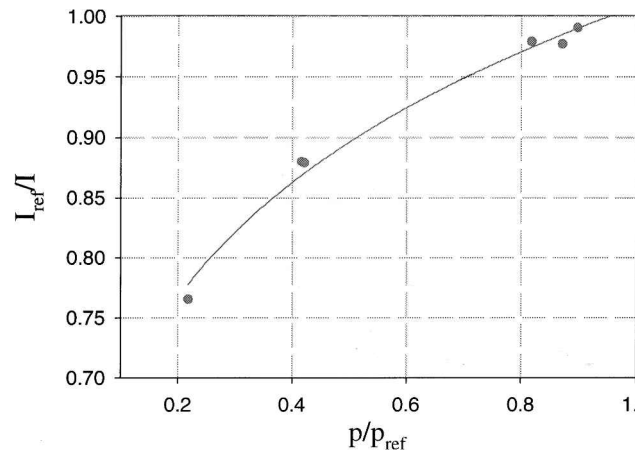


Fig. 8 In situ calibration curve of AA-PSP with the supporting matrix thickness of 9 μm; adsorption controlled model used to determine calibration constants.

Step Change of Pressure

Figure 7 shows luminescent intensity signals of AA-PSP with the supporting matrix thickness of 9.0 μm. Initial driven and driver pressures, p_1 and p_4, were 25.6 and 100 kPa, respectively. The luminescent intensity signals are shown in a normalized scale. The step change of luminescent intensity was converted to pressures through the in situ calibration curve shown in Fig. 8 because in situ calibration takes into account temperature effects.

Figure 9 shows pressure signals of AA-PSP and the pressure tap, showing the theoretical pressure jump due to incident and reflected normal shock waves. Because the unsteady reference transducer has a natural frequency of 13 kHz, signals were low-pass filtered at 5 kHz to obtain quasi-steady pressure signals. The AA-PSP results show a sharp pressure rise after the incident and reflected shock waves.

Response Time Measurement

A step change of pressure from an incident normal shock wave was used for response time calculation. Pressure signals were normalized to account for small pressure differences during individual runs. Four different thicknesses of the porous supporting matrix were considered for AA-PSP response time measurements: 4.3, 9.0, 13.2, and 27.2 μm. These AA-PSPs are denoted as AA-PSP$_4$, AA-PSP$_9$, AA-PSP$_{13}$, and AA-PSP$_{27}$, and normalized pressure

Table 1 List of response times and thicknesses of supporting matrix

PSP	Thickness, μm	Response time, μs
AA-PSP$_4$	4.3	34.8
AA-PSP$_9$	9.0	70.9
AA-PSP$_{13}$	13.2	80.0
AA-PSP$_{27}$	27.2	102
TLC-PSP	152	65.1
PC-PSP	~50	349
Polymer[a]	~60	172,000

[a]Polymer PSP (conventional PSP).

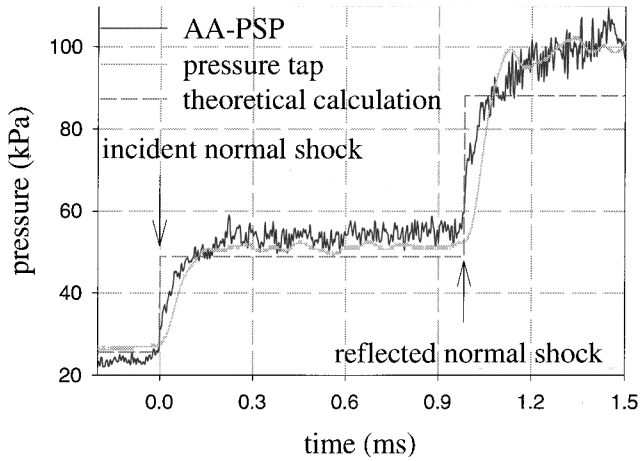


Fig. 9 Pressure data obtained from AA-PSP with the supporting matrix thickness of 9 μm and pressure tap compared with theoretical calculation.

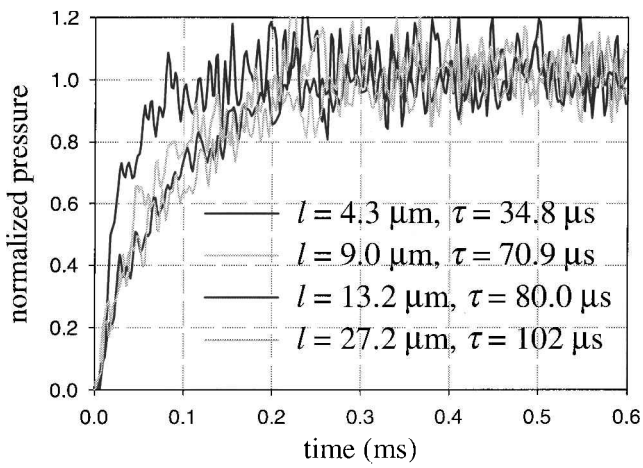
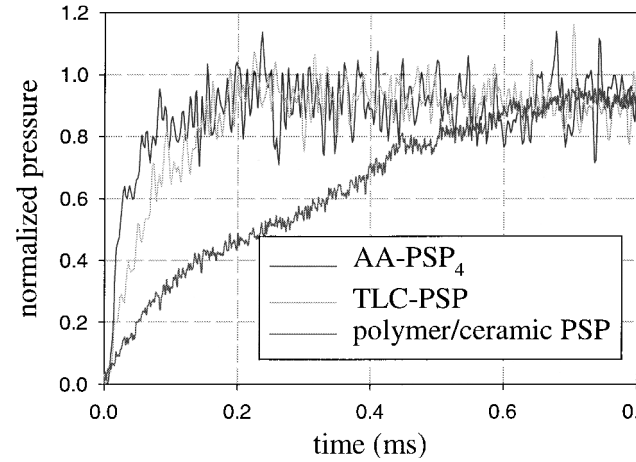


Fig. 10 Normalized pressure of AA-PSPs with different matrix thicknesses *l* for response time measurements.

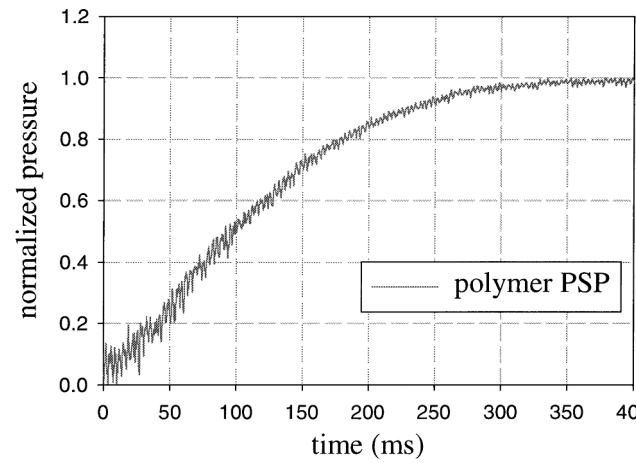
signals for these PSPs are shown in Fig. 10. Response times of these PSPs were determined from the first-order system[4] and are listed in Table 1. Response time τ increases as the matrix thickness increases. The fastest response time of 34.8 μs was obtained from AA-PSP with the matrix thickness of 4.3 μm.

Comparison of PSPs with Different Binders

Figures 11a and 11b compare a step change of pressure obtained from AA-PSP, TLC-PSP, PC-PSP, and conventional PSP. Pressures are shown in a normalized scale. The conventional polymer PSP could not respond fast enough to measure the first step change. Therefore, oxygen was sprayed onto this PSP to measure a slow response time. Hence, the conventional PSP was normalized from the minimum to the maximum pressures (1 atm). Note that the timescale of Fig. 11b is 500 times longer than that of Fig. 11a.



a) Comparison with porous PSPs



b) Comparison with conventional PSP

Fig. 11 Comparison of AA-PSP with the supporting matrix thickness of 4 μm (AA-PSP$_4$) to TLC-PSP, PC-PSP, and polymer PSP.

All of the response times are listed in Table 1. The response times of AA-PSP and TLC-PSP were on the same order of magnitude. PC-PSP showed slower response even though the thickness of the porous supporting matrix was thinner than that of TLC-PSP. This is because PC-PSP uses a small amount of polymer. Scorggin[12] optimized the polymer content of this PSP and measured 60-μs response time. TLC-PSP could give a fast response. However, the thickness of the porous supporting matrix cannot be controlled so that the response cannot be optimized by reducing the thickness. The matrix material changed the response time more than the matrix thickness. The response time of the PSP with polymer matrix was the slowest response time, which is on the order of hundreds of milliseconds. On the other hand, porous PSPs have response times on the order of tens of microseconds.

Conclusions

Using anodized aluminum as a porous supporting matrix, a fast response time of 34.8 μs was obtained with a matrix thickness of 4.3 μm. The matrix thickness of the anodized aluminum influences the response time. A thinner matrix thickness gives a faster response time.

The temperature increase on the paint surface due to the shock waves was observed by using a TSP with rhodamine-B on anodized aluminum. The temperature increased ~2 K as the incident normal shock wave passes by. This gives a 2.3% pressure uncertainty for a PSP with bathophen ruthenium on anodized aluminum when using a priori calibration.

AA-PSP results agreed with pressure tap measurements and theoretical calculations through in situ calibration, which takes into account the temperature dependency of AA-PSP. Porous PSP samples have response times on the order of tens of microseconds, which

are three orders of magnitude faster than PSP with a polymer matrix. The matrix material greatly changes the response time.

Acknowledgments

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