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Characterization of multi-dye pressure-sensitive microbeads

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The response times of pressure-sensitive particles to passing shockwaves were measured to investigate their ability to accurately determine pressure changes in unsteady flows. The particles tested were loaded with novel pressure-sensitive dyes such as Pt (II) meso-tetra(pentafluorophenyl)porphine, Pt(II) octaethylporphine, bis(3,5-difluoro-2-(2-pyridyl)phenyl-(2-carboxypyridyl))iridium III, and iridium(III) bis(4-phenylthieno[3,2-c] pyridinato-N,C2')acetylacetonate. For this work, porous silicon dioxide pressure-sensitive beads (PSBeads) were used. Two synthetic procedures were used to fabricate the particles. In the first, a one-step method loaded dyes during the synthesis of microbeads, in the second a two-step method synthesized the microbeads first, then loaded the dyes. The shock tube facility was used to measure the response times of microbeads to fast pressure jumps. The study involved testing multiple luminophors loaded in microbeads with various size distributions. Response times for the silica-based microbeads ranged between 26 μs and 462 μs (at 90% of the amplitude response), which are much faster than previously reported polystyrene-based microbead response times, which range from 507 μs to 1582 μs (at 90% of the amplitude response) [F. Kimura, M. Rodriguez, J. McCann, B. Carlson, D. Dabiri, G. Khalil, J. B. Callis, Y. Xia, and M. Gouterman, "Development and characterization of fast responding pressure sensitive microspheres," *Rev. Sci. Instrum.* **79**, 074102 (2008)]. © 2013 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4824699>]

I. INTRODUCTION

Pressure-Sensitive Paints (PSPs) have allowed for the non-intrusive global measurement of pressure on aerodynamic surfaces.²⁻⁵ The use of PSP is becoming more common in large transonic tunnels, with production systems in use in several facilities such as AEDC,⁶ ARA,⁷ TsAGI,⁸ and DLR.⁹ Fast-responding PSP has been used in unsteady aerodynamic applications, such as airflow over rotor blades.¹⁰ An excellent review of unsteady aerodynamic applications of PSP was presented by Gregory *et al.*¹¹

PSP contains oxygen-sensitive molecules that are held within an oxygen permeable polymer binder. When illuminated with absorbing wavelengths, the excited molecules release part of their energy as photons. Surrounding oxygen molecules can absorb these emitted photons. Hence, the luminescence observed is inversely proportional to the surrounding oxygen concentration within the surrounding atmosphere; this well-known phenomenon is called the luminescence quenching. As concentration of oxygen in the air is proportional to pressure, PSPs can be used to accurately measure pressure. In implementation, light sources with the appropriate wavelengths are used to illuminate the de-

sired surface, and CCD cameras are used to measure the light emissions from the PSP-painted surfaces. This methodology has been successfully used in wind-tunnel applications⁵⁻¹² and is now commercially available.¹³ Details of this methodology can be found in literature.⁵⁻¹⁴

The success of the PSP methodology has inspired the pursuit of globally measuring pressure within the fluid flow. Abe *et al.*¹⁵ used pressure-sensitive particles to qualitatively show that pressure variations could be observed within the flow. More recently, dual luminophor polystyrene microbeads (1–5 μm diameters) have been developed by our groups. That allows for self-referencing pressure-sensitive microbeads.¹⁶ We have further demonstrated that these microbeads can be used to measure pressure and temperature simultaneously.¹⁷

Although these microbeads can accurately measure pressures and temperatures, it is essential to quantify the response time to changing pressures and temperature to determine whether the microbeads can adequately perform their function in unsteady environments. The microbeads must respond sufficiently fast to capture rapid changes in pressures and temperatures if they are to be used in conjunction with unsteady flow fields alongside steady aerodynamic regions. The response time of PSPs has been described as being dependent on three important parameters: Luminescent lifetime of the luminophor, the oxygen diffusivity, and thickness of matrix layer. Kimura *et al.*¹⁸ found the lifetime in a previous

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study where it was shown that the scope of the luminophors' lifetime expands from 1 μs to 50 μs . The estimation of the 99% rise time of a thin PSP layer can be expressed as

$$\tau_{99\%} = \frac{12 \times L^2}{\pi^2 \times D}$$

and

$$\tau_{99\%} = \frac{3 \times d^2}{4 \times \pi^2 \times D}$$

for a microbead, where L is the thickness of the layer, d is the diameter of the bead, and D is the oxygen diffusion coefficient of the matrix being used.^{19–21} Hence, to develop a fast responding microbead, a compromise would have to be made between the thickness of the layer and the oxygen diffusion coefficient. Kimura *et al.*¹ reported that the response times for 2 μm diameter polystyrene microbeads are estimated to range from 9.8 ms to 27.6 ms, which would be too slow for desired applications, such as measuring pressure changes in turbulent flows. This led to initial research involving the synthesis of a new type of microbead using a highly porous silicon dioxide structure containing novel pressure-sensitive osmium complexes, with a much faster response time (Kimura *et al.*¹). While successful, the osmium-based silicon dioxide microbeads had a very low signal-to-noise ratio. Towards this end, the purpose of this paper is to exhibit the improvements upon the synthesis of silica microbeads with higher signal-to-noise ratios using new multi-loaded dyes, while maintaining sensitivity and fast response times.

II. EXPERIMENTAL METHOD

A. Pressure-sensitive microbeads

1. Chemicals and materials

Cetyltrimethylammonium bromide (CTAB), ammonium hydroxide ($\text{NH}_3 \cdot \text{H}_2\text{O}$, 28% NH_3 in H_2O), tetraethyl orthosilicate (TEOS), methanol, ethanol, and acetone were all purchased from Sigma-Aldrich. Deionized water with a resistivity of 18.2 $\text{M}\Omega \text{ cm}$ was used for all the syntheses. The microbeads are loaded with three different luminescent dyes. The first type is from a family of pressure-sensitive dyes composed of Pt(II)octaethylporphine (dye A), Pt(II) meso-tetra(pentafluorophenyl)porphine (dye B), and bis(3,5-difluoro-2-(2-pyridyl)phenyl-(2-carboxypyridyl)iridium III (dye D). The respective emission wavelengths of the pressure dyes are 650 nm, 650 nm, and 500 nm. The second type of dye corresponds to a reference dye, Coumarin 500 (dye H) or Mg(II)meso-tetra(pentafluorophenyl)porphine (dye J). Their emission wavelengths are 530 nm and 650 nm, respectively. The emission intensities of both reference dyes are insensitive to pressure and temperature changes. The third dye incorporated in the microbeads is the temperature-sensitive dye Eu(III)thenoyltrifluoroacetate (dye E) with a 615 nm emission wavelength. Dyes A, B, J are from Frontier Scientific, dyes C, D, H are from Luminescence Technology Corporation and dye E is from Gelest. All chemicals were used as received. These materials are used

to prepare mesoporous silica microbeads per the synthesis methods described below, where the mesopores are estimated to be 1–2 nm in size. The CTAB molecules serve as templates for the generation of these mesopores during the synthesis process.

2. Synthesis of mesoporous silica microbeads loaded with different dyes using a one-step method

In a typical synthesis, 0.4 g of CTAB, 167 ml of methanol, 30 ml of water, and 11 ml of $\text{NH}_3 \cdot \text{H}_2\text{O}$ were mixed in a 250 ml flask under magnetic stirring. Meanwhile, different combinations of dyes with specific amounts were dissolved in 1.5 ml of acetone. The dyes were then introduced into the mixture, followed by rapid injection of 0.6 ml of TEOS using a pipette. The reaction was allowed to proceed at room temperature for 24 h. The resultant functionalized mesoporous silica microbeads were collected by centrifugation and washed with water three times.

3. Synthesis of mesoporous silica microbeads loaded with different dyes using a two-step method

The two-step procedure was composed of the synthesis of mesoporous silica beads utilizing a seed-mediated process, and the loading of dyes into the as-obtained silica beads. In a typical synthesis, 100 mg of CTAB, 40 ml of methanol, 7.5 ml of H_2O , and 3 ml of $\text{NH}_3 \cdot \text{H}_2\text{O}$ were placed in a 100 ml flask, followed by the introduction of 25 μl of TEOS to generate primary silica seeds. After the reaction was proceeded for 1.5 h, 2.4 ml of TEOS was injected into the solution at the rate of 0.4 ml/h with the help of a syringe pump to start the growth. The reaction was allowed to proceed at room temperature under magnetic stirring for 24 h. The resultant mesoporous silica microbeads were collected by centrifugation and washed with ethanol three times. The products were redispersed in 5 ml of ethanol for further use. For the loading of dyes, different combinations of dyes with specific amounts were dissolved in 1.5 ml of acetone at room temperature, and then introduced into 1.5 ml of as-obtained silica beads. The mixture was ultrasonically dispersed for 1 h and then magnetically stirred overnight. The final products were collected by centrifugation and washed with water three times.

Additionally, larger size silica microbeads were used in the fabrication process. For these $\sim 14 \mu\text{m}$ in diameter hollow glass spheres, Sphericel 110P8, from Potters Industries were used and loaded with dyes. When reporting results in Section III, the synthesized silica microbeads will be identified with an asterisk (*).

Several iterations of different dye concentrations were synthesized. For example, microbead samples were loaded with different ratios of the B:E:H dyes. Over 60 samples of microbeads were evaluated for spectral characteristics and response time to pressure jumps. Test samples were made by drop-casting 100 μl of water suspension ($\sim 10\%$ solids) of the microbeads onto the surface glass slide of 3 cm by 1 cm. The samples were dried in an oven set at 70 $^\circ\text{C}$.

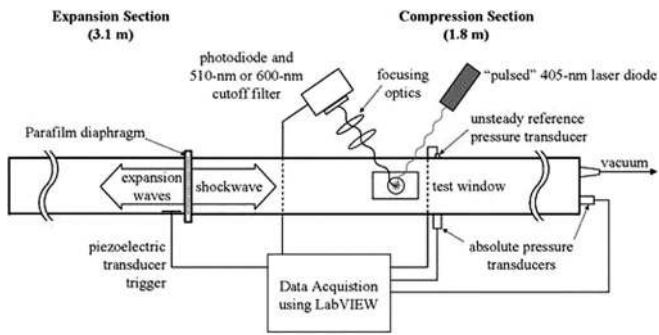


FIG. 1. Schematic of the shock tube. Reprinted with permission from F. Kimura, M. Rodriguez, J. McCann, B. Carlson, J. Callis, D. Dabiri, and G. Khalil, *Rev. Sci. Instrum.* **79**(7), 074102 (2008). Copyright 2008 American Institute of Physics.

B. Shock tube

A shock tube was used for this study to measure the response time of the microbeads to rapid pressure jumps. Other techniques for response time have been studied²² though the shock tube presented a reliable method for determining response times of pressure rises at the microsecond scale. Gregory and Sullivan²² characterized the response time of pressure sensitive paints using a fluidic oscillator and found that the paints responded faster to pressure decreases than to pressure increases. Thus, this research focused on pressure rises as the limiting factor to response time. The shock tube setup consisted of a square aluminum tube with walls 0.64 cm thick, with a cross section of 3.9 cm by 3.9 cm. The schematic of the testing facility is presented below in Figure 1.

The shock tube was assembled by two main sections, a 3.1 m long expansion (driver) chamber, and a 1.8 m long compression (driven) chamber. A diaphragm was positioned at the connection between the two sections that burst when the pressure difference between both chambers was high enough, causing a shockwave to propagate down the compression section. Further details on the theory and testing of shock tubes can be found in textbooks²³ and in Kimura *et al.*,¹ respectively. The diaphragm was made of Parafilm[®] M (127 μm in thickness, Bemis flexible Packaging, Neenah, WI) and the thickness was varied by modifying the number of layers of Parafilm. A configuration of six layers of Parafilm was used as the pressure decreased from 100 kPa to 4 kPa, a pressure ratio obtained of 1/25.

The pressure difference was established in one of two ways: A vacuum pump was used to directly pump out the air in the compression chamber or used to generate low pressure in a large tank, which was then connected to the compression chamber. The motivation for using the latter was to reduce the wait time for the vacuum pump to lower the pressure directly in the compression section. Two 1.9 cm by 3.8 cm test windows, on which the microbeads test samples were mounted, were positioned 0.58 m downstream of the diaphragm. Samples were placed on this top window of the shock tube, oriented face down, in order to reduce the optical interference of the shockwave with the illuminating samples, but still maintain a direct face on the passing shockwave.

Unsteady and steady pressures were measured 0.12 m downstream of the test window using a high sensitivity

dynamic pressure sensor with a 90% rise time of 2 μs (model 211B5, Kistler Instruments, Amherst, NY) attached to a power supply coupler (model 5114, Kistler Instruments, Amherst, NY) for the unsteady pressure measurements. For the steady pressure measurements, an Omega PX236 series pressure transducer was used. The pressure transducers were positioned in the tube flush with the walls to not interfere with the flow. The stated accuracy of the steady pressure transducers were 0.25% of the full scale.

C. Luminescence and data acquisition

The detection system was composed of a 405 nm continuous laser light used to excite the microbeads. The test sample was attached to the top window such that the microbeads were facing down in direct contact to the shock wave. The light emitted by the test sample was focused on a Photomultiplier (PMT Hamamatsu R928) fitted with a band-pass filter at the appropriate wavelength. The PMT had a 2.2 μs rise time and a gain of 10^7 for an applied voltage of 1000 V. Additionally, a band-pass filter was positioned in front of the laser to reject any other lines. Furthermore, the data from each transducer used were processed by Signal Express Labview through the National Instruments data acquisition board (BNC-2120) connected to the computer, sampling at 100 ks/s.

Once the sample was placed onto the window, the vacuum pump pumped down the downstream section of the shock tube until the diaphragm burst, all the while the pressure and intensity data were being recorded. The data obtained was exported, plotted, and processed with Excel to calculate the response time of the tested microbeads based on a 63.2% and 90% rise time of the intensity change. Although the responses of PSPs have been shown to follow exponential models, it is more useful to characterize the response time as a percent increase in rise time of the intensity change¹ rather than multiple time constants associated with the multi-exponential models. More recent studies have also concluded that the 90% rise time is a more valuable representation of the response time.²⁴

III. RESULTS AND DISCUSSION

A. Emission spectra and initial results

At first, the emitted light spectrums of the silica microbeads were examined to determine whether the particular samples of silica microbeads were sufficiently illuminating at the wavelengths of each dye. An example of several silica microbeads is shown in Figure 2 which illustrates the spectral response of the particular set of silica microbeads.

In order to demonstrate that our procedures in determining response time were sufficient, initial tests of silica BEH* microbeads (again, the * denoting silica microbeads synthesized by the Xia research group rather than commercial silica microbeads) were performed and the signals for each of the three dyes incorporated into the microbeads was captured. Figure 3 shows these microbeads' response to a pressure jump along with the dynamic pressure transducer.

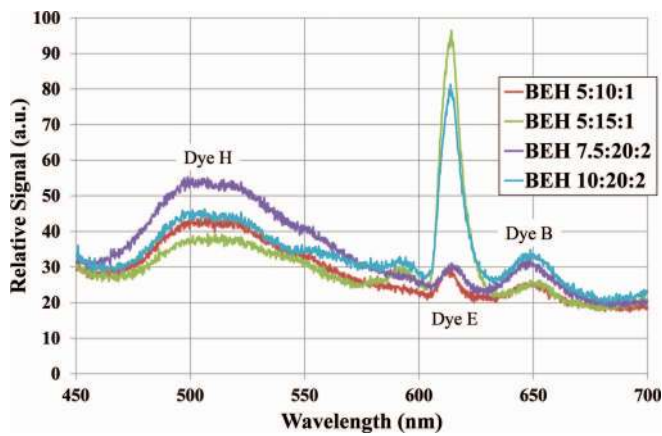


FIG. 2. Emission spectra of several silica microbeads excited with a 365 nm LED showing the wavelengths of the different dyes loaded into the silica microbeads.

The initial pressure of the driven and driver chamber, P_1 and P_4 , were, respectively, 4 kPa and 100 kPa with a six-layered Parafilm diaphragm as stated in Sec. II. The dye intensity data presented in Figure 3 exhibits a relative magnitude of the signals in order to prove that the variation of the reference dye and the temperature signals are very small compared to the change of emitted light intensity by the pressure dye. The decrease in signal of the differential pressure transducer after the shock is seen as a slight rise in pressure dye, although this is not a part of the response time. As a result, this proved that the testing facility along with the microbeads tested was reliable as a means for measuring pressure change within a flow field.

In this research, the signal-to-noise ratio was a significant concern in the way that it needed to be high enough to prove the efficiency of the microbeads tested as a tool for measuring pressure within an unsteady fluid flow. A specific criterion for a signal-to-noise ratio of approximately 16 was used to assess whether the microbeads would be adequate for testing. Additionally, the data presented in this study were ten point averages of the raw data processed in LabView. The average of the raw signal was performed in order to reduce the noise

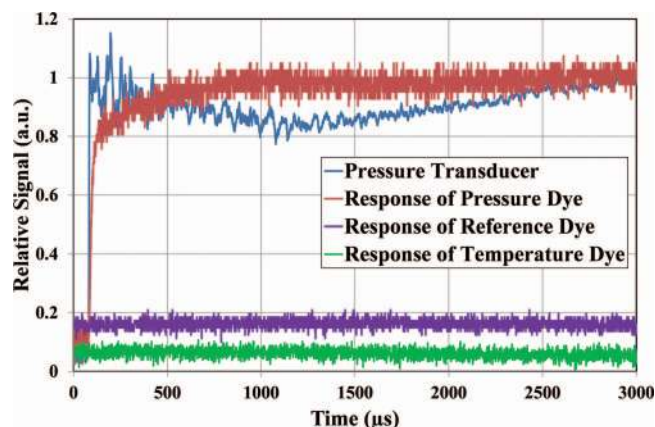


FIG. 3. Plot of the intensity changes of Silica BEH* (10:25:0.5) microbeads excited with a continuous 405 nm laser light for each dye incorporated into the microbeads as well as the dynamic pressure sensor.

TABLE I. Calculated response times for samples of microbeads tested within the shock tube facility.

Sample	Fabrication method	Dyes ratio	Response time (μs)	
			63.2%	90%
Silica BEH*	One step ^{16,25}	BEH* (5:50:0.05)	20	28
Silica BEH*		BEH* (10:50:0.05)	180	268
Silica BEH*		BEH* (10:50:0.05)	144	266
Silica BEH*		BEH* (10:50:0.5)	112	252
Silica BH*	Two steps	BH* (10:0.5)	26	60
Silica BEH		BEH (5:25:0.5)	24	44
Silica BEH*		BEH* (5:25:0.5)	26	50
Silica BEH*		BEH* (10:25:0.5)	30	52
Silica BEH*		BEH* (10:25:0.5)	56	80
Silica BEH*		BEH* (5:25:0.25)	24	44
Silica BEH*		BEH* (10:25:5)	40	84
Silica BEH*		BEH* (5:25:0.5)	54	78
Silica BEH*		BEH* (5:10:0.5)	64	90
Silica BEH*		BEH* (5:15:1)	28	42
Silica BEH		BEH (15:20:2)	46	78
Silica BEH		BEH (10:20:20)	28	62
Silica BEH*		BEH* (5:15:1)	24	52
Silica BEH*		BEH* (7.5:20:2)	24	52
Silica BEH*		BEH* (10:20:2)	30	64
Silica BEH		BEH (10:20:1)	118	310
Silica DJ*		DJ* (10:1)	150	462
Silica BE		BE (10:20)	16	26
Silica BE		BE (20:20)	20	32
Polystyrene AEH	One step ^{16,25}	AEH	276	507
Polystyrene AEH		AEH	716	1582

that still persists in addition to smoothing out the curve for better response time calculations.

B. Response time results

All the data obtained were analyzed in order to find the response times of each sample as explained in Sec. II A summary table of the testing and response time results is presented in Table I. The range of response times at 90% rise time due to the shock wave spreads from 26 μs to 1582 μs . For reference, the response times of polystyrene microbeads (following previous procedures²⁵ are also included, which show that they are the largest response times, which is consistent with previous results.¹ The polystyrene and silica microbead response time comparison is also shown in Figure 4.

While Figure 4 shows that the two-step fabrication method of the silica microbeads has a faster response time than the polystyrene microbeads, it is also seen in Table I that the two-step fabrication method also consistently demonstrates faster response times than the one-step fabrication method. The BEH* microbeads fabricated using the one-step method has average ($n = 4$) 63.2% and 90% response time values of 114 μs and 204 μs , respectively, with standard deviations of 69 μs and 117 μs . However, the BEH and BEH* (both commercial silica microbeads and microbeads synthesized during the fabrication process) fabricated using the

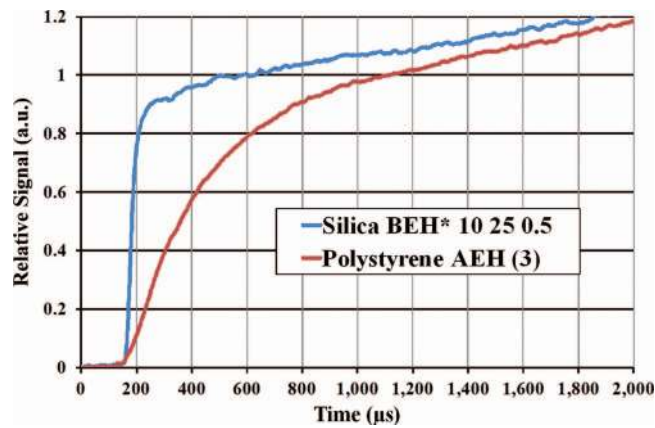


FIG. 4. Plot of the response time of a silica two-step fabrication method BEH microbeads test and a polystyrene AEH microbeads test excited with a continuous 405 nm laser light showing significantly slow response time from the polystyrene microbeads.

two-step method has respective averages of $41 \mu\text{s}$ and $79 \mu\text{s}$, with standard deviations of $25 \mu\text{s}$ and $66 \mu\text{s}$. Therefore, the average response time using the two-step fabrication method was approximately a third of that using the one-step method, with significantly lower deviations. Lastly, the silica BE microbeads were the final and best effort to create microbeads using the two-step method and resulted in the fastest response times, with an averaged 63.2% and 90% rise time of $18 \mu\text{s}$ and $29 \mu\text{s}$, respectively, with $2.8 \mu\text{s}$ and $4.2 \mu\text{s}$ standard deviations, respectively.

A plot of several silica microbeads tests is presented in Figure 5, which illustrates the consistency of fast responding microbeads using the two-step fabrication method. Correspondingly, Figure 6 presents a plot of a one-step fabrication method silica microbeads test and a two-step fabrication method test further showing the slow response time of the one-step method compared to that of the two-step method. Furthermore, the microbeads loading did not seem to affect the response time, as Table I shows no distinct relationship between the dye loading and better response times.

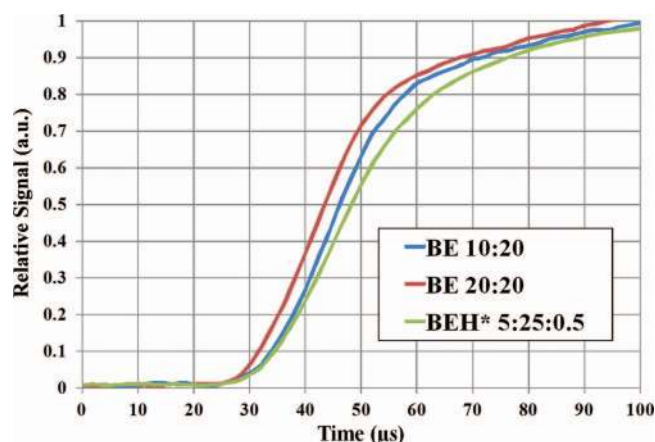


FIG. 5. Plot of the response time of fastest BE (two-step) and BEH (two-step) microbeads excited with a continuous 405 nm laser light showing consistency of fast responding two-step fabrication method.

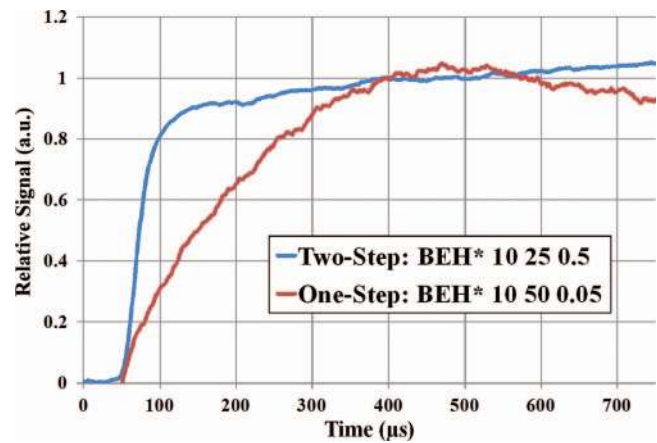


FIG. 6. Plot of the response time of a one-step BEH microbeads test and a two-step BEH microbeads test excited with a continuous 405 nm laser light comparing the two different fabrication methods.

One additional investigation involved testing some of the microbead samples using a different illumination pattern. Instead of illuminating the test sample with a laser spot that ranged from 5 mm to 10 mm in diameter, a 1 mm thick laser line was created, using a cylindrical lens, to illuminate the test sample perpendicular to the direction of the shock propagation. As the shock transit time over the laser line is much quicker than over the laser spot, the motivation for this change was to investigate whether the measured response time would be more precise. The results of these tests did not show improvements in the response time, therefore suggesting that the laser line illumination was not necessary, and that a laser spot was sufficient.

IV. CONCLUSIONS AND FUTURE WORK

The response times of different pressure-sensitive multi-dyed microbeads to passing shock waves were measured. The silica-based microbeads exhibited response times ranging between $26 \mu\text{s}$ and $462 \mu\text{s}$. The majority of the silica-based samples showed adequate response times for use in unsteady flow investigations. The particular microbeads tested exhibited high signal-to-noise ratios as well as high sensitivity while maintaining their fast response times. The data revealed that the most significant contribution to response time is the fabrication method of the microbeads, particularly since the two steps method of fabrication consistently produced fast responding microbeads. The dye loading of the microbeads showed no correlation to the response times. Therefore, the fabrication and method of incorporating the dyes into the microbeads is the underlying contributor to the response time, not the amount of each dye. Future work will report on temperature response time measurement methods as well as their results.

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