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Application Note

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Materials

Method for Measuring the In-flight Velocity of Microparticles Using Mechanoluminescent Materials

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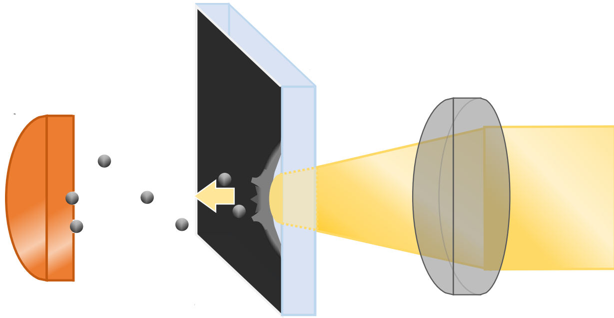

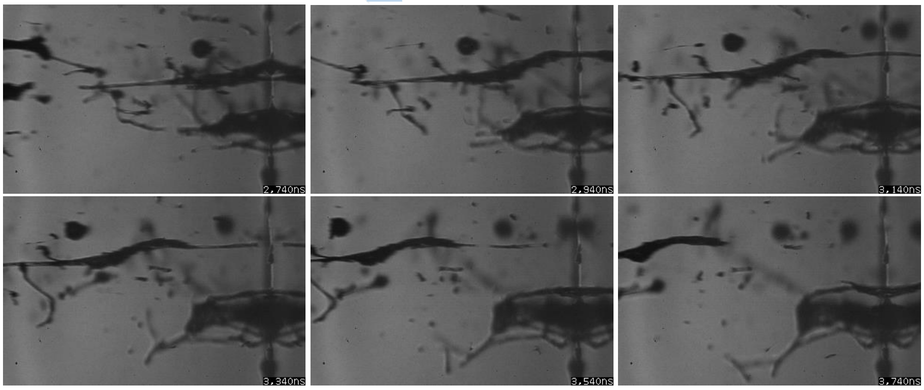

Materials

■ Abstract

The Laser Induced Particle Impact Test (LIPIT) was developed as a small-scale impact testing technology. In this article, high-velocity in-flight particles were recorded using the Hyper Vision™ HPV™-X2 high-speed video camera with this technology. An in-flight velocity measurement method using mechanoluminescent materials was also developed, and the usefulness of the method was confirmed by comparing it with the measurement results using the HPV-X2. In addition, a surface-modifying method for metal materials was investigated. It was found that the microstructure of metal materials can be made finer through the plastic deformation caused by high strain and high strain rate impacts, leading to material strengthening.

1. Introduction

The technique of performing high-velocity particle experiments has been widely utilized in the aerospace industry for material strength research and surface modification processing. Conventional methods require large-scale experimental equipment; however, the LIPIT method, which enables high-velocity impacts of microparticles even with compact equipment, has been proposed recently. LIPIT uses laser ablation driven by a pulsed laser as the driving force, allowing micro-nanoparticles to be ejected at a velocity of several kilometers per second[1, 2).

Flight velocity measurements are important in the study of impact materials and the performance evaluation of experimental technologies. In a previous report[3)], in-flight particles were recorded using the HPV-X2, and their velocities were calculated from the images. In this article, a method for measuring flight velocity using a mechanoluminescent material that emits light due to micro-deformations, such as elastic deformation, was developed, and the validity of the method was evaluated based on the velocity measurement results from the HPV-X2. Furthermore, the potential of using LIPIT as a new surface modification technology was investigated.

2. Laser Induced Particle Impact Test (LIPIT)[4)]

In the method described in this article, laser ablation was used as the driving force to eject microparticles at high velocity. The ejected microparticles that hit the target specimen cause indentations due to plastic deformation. The depth of the indentation enables evaluation of the deformation strength at high strain rates. Fig. 1 is an overview of the LIPIT used in this article. The pulsed laser beam emitted from the Nd:YAG laser is directed onto the emitting stage through a condenser lens. The emitting stage consists of a transparent constraining glass and a black energy-absorbing layer (EA layer), which expands rapidly in volume due to the ablation phenomenon. Microparticles scattered on the surface of the energy-absorbing layer are emitted directly toward the target materials due to the instantaneous large deformation. To measure the in-flight velocity of these particles, visualization was conducted using the high-speed video camera shown in Fig. 2 (HPV-X2).

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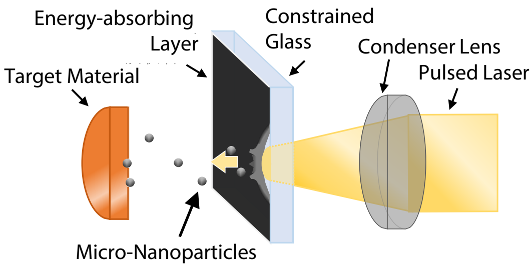

Fig. 1 Laser Induced Particle Impact Test (LIPIT)

Fig. 2 High-Speed Video Camera Hyper Vision™ HPV™-X2

3. Particle Velocity Measurement Using the HPV-X2 High-Speed Video Camera

The behavior of in-flight microparticles was recorded using the shadowgraph method. The recording setup is shown in Fig. 3, and the recording equipment is listed in Table 1. At a recording speed of 5 Mfps, the trajectory of zirconia particles (30 µm in diameter, $30ZrO_2$) was clearly captured, and the recording was successful (Fig. 4). The captured images revealed that the scattered EA layer flew ahead of the particles and that the leading particle flew at the highest velocity. These experiments were conducted using microparticles of various sizes and weights, and the respective flight velocity were calculated. As a result, super high-velocity particles that reached a maximum velocity of 800 m/s were recorded successfully.

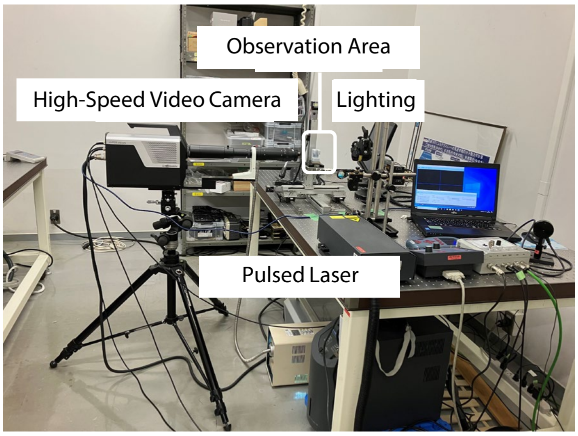

Fig. 3 Recording with a High-Speed Video Camera

Table 1 Recording Equipment

High-Speed Video Camera	:	HPV-X2
Microscope	:	Z16 APO
Lighting	:	Cavilux

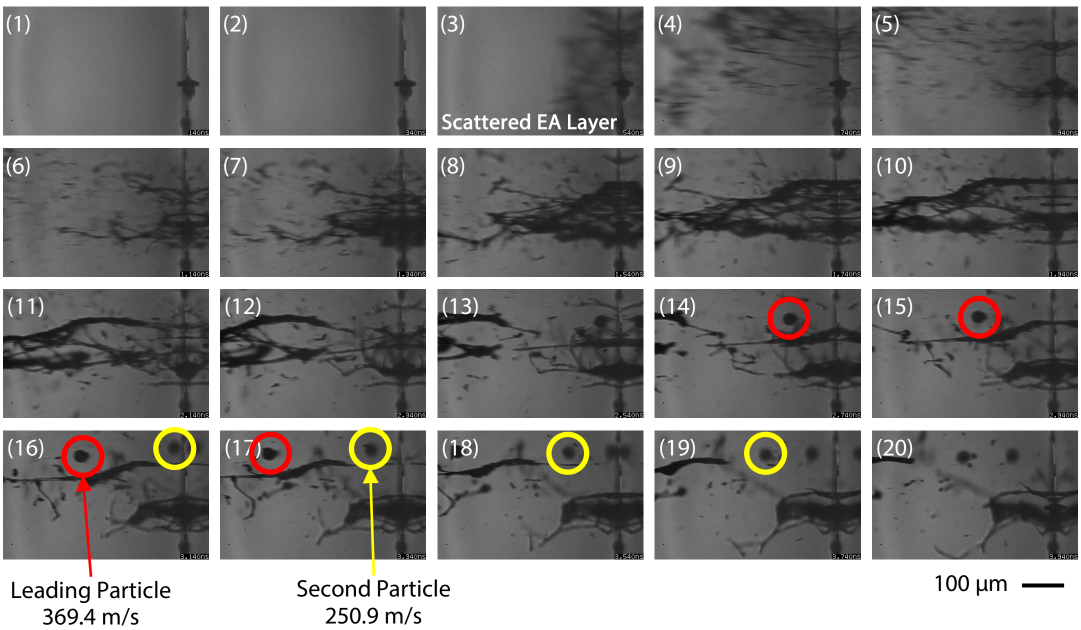

Fig. 4 Particle Velocity Measurement Results with a High-Speed Video Camera (The Recording Interval between Images is 200 ns)

4. Particle Velocity Measurement Using Mechanoluminescent Materials

The mechanoluminescent material (Mechanoluminescence: ML) used in this study is an inorganic phosphor that releases energy in the form of light when subjected to mechanical stimulation. It luminesces even when the deformation is small such as in the elastic deformation region, and can emit light repeatedly. Making use of this property, the luminescence was measured when the particles emitted by LIPIT collided with the ML using the particle velocity measurement method shown in Fig. 5. The mechanoluminescent material, which has an increased luminescence when excited by ultraviolet radiation, was irradiated with UV light for 30 seconds before the test. The ML material used in this test is a mechanoluminescent composite resin made by mixing a two-component epoxy adhesive with mechanoluminescent powder, which was applied to aluminum foil and is referred to as the 'ML sensor.' The time until the luminescence intensity increased was recorded because the intensity rises when the emitted particles collide with the ML sensor. The photons were converted into electric pulse signals in the photomultiplier tube, and the amount of luminescence was measured by outputting to an oscilloscope. The acquired output waveform is shown in Fig. 5. A pulse of about 3 V was counted as one photoelectron. The photoelectron pulses measured by the oscilloscope originate from three factors: the phosphorescence (noise) of the luminescent material under no load, luminescence due to the collision with the scattered EA layer, and luminescence from particle collisions, are all combined. Therefore, to obtain the particle velocity, it is necessary to separate the luminescence due to particle collisions from that due to other sources.

Therefore, data was collected under the conditions of 'no particles (only the scattered EA layer)' shown in Fig. 6(a) and 'with particles' shown in Fig. 6(b). These two conditions were treated as one set of experiments, and the accumulated photoelectrons generated from the particle ejection time to each time point were plotted, resulting in Fig. 6(c). From this graph, it can be seen that the initial rising portion where the two curves overlap is due to the luminescence caused by the scattered EA layer, which travels faster than the particles. This is also supported by the images captured by the high-speed video camera shown in Fig. 4. Subsequently, the two curves diverge, resulting in a difference in luminescence, which is believed to be due to particle collisions. Therefore, this divergence point was considered to be the collision time of the leading particle. The average particle velocity obtained from this method was compared with the values measured by the high-speed camera, and the results are shown in Fig. 7. Since the result are in close agreement for each particle, this indicates that the particle measurement method using the ML sensor developed here was successful. A detailed validation of this velocity measurement method is described in references 4) and 5).

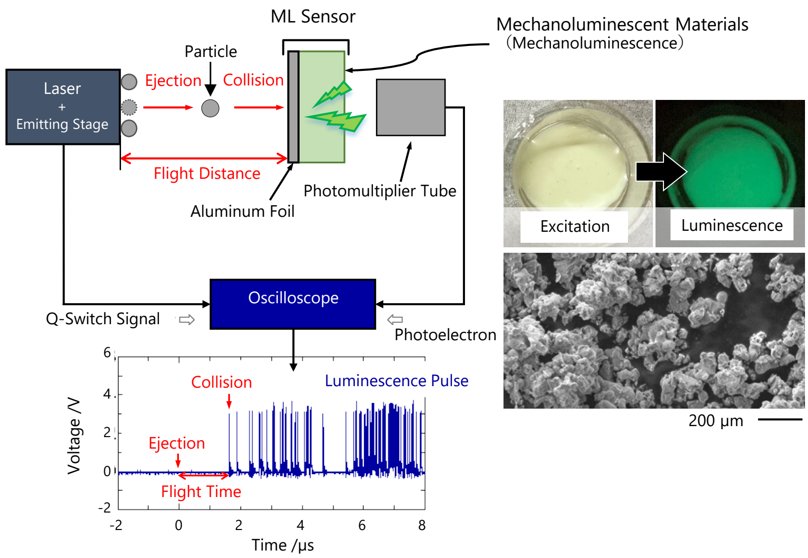

Fig. 5 Schematic Diagram of the Particle Velocity Measurement Method Using Mechanoluminescent Materials

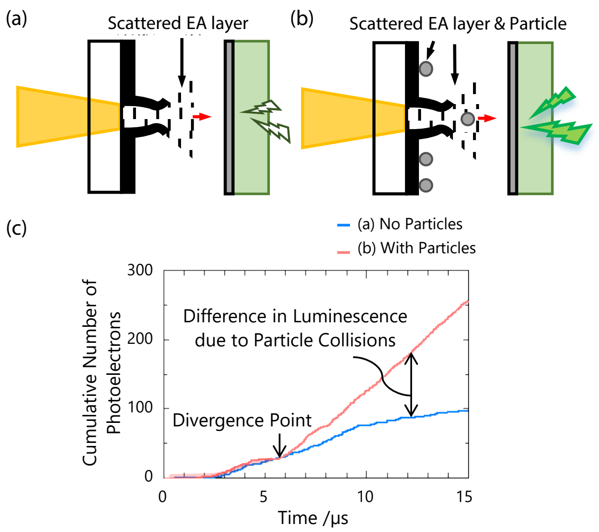

Fig. 6 Separation of Particle Collisions and Other Luminescence to Calculate Particle Velocity (a) No Particles, (b) With Particles, (c) Cumulative Number of Photoelectrons at Each Time (0 is the Ejection Time)

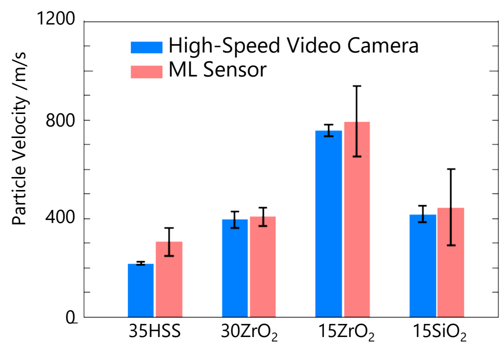

Fig. 7 Comparison of Particle Velocity Measured by High-Speed Video Camera and ML Sensor

5. LIPIT Application Example (Microstructure of Metal Materials)

LIPIT was conducted on pure copper specimens using $15ZrO_2$ and $30ZrO_2$. The results of the observation of the impact indentations are shown in Fig. 8. Fig. 8(a) shows the SEM images of the impact indentations obtained from $15ZrO_2$. In contrast, Fig. 8(b) shows examples of the indentation cross-sectional shapes obtained from a scanning laser microscope for $30ZrO_2$. The average depth of the indentations is approximately 7.4 µm for $15ZrO_2$ and approximately 11.6 µm for $30ZrO_2$. In addition, when calculating the spherical strain by excluding the depth from the diameter of each indentation, the values were 0.6 for $15ZrO_2$ and 0.4 for $30ZrO_2$, indicating that $15ZrO_2$, which collides at a higher velocity, generates greater plastic strain.

To achieve greater spherical strain, LIPIT with $15ZrO_2$ was applied to pure iron at a collision velocity of 750 m/s, and the cross-section of the resulting impact indentation was extracted to obtain a SIM image. For comparison, the cross-sections of the indentations produced by a quasi-static indentation test (Dynamic Ultra-Micro Hardness Tester: DUH™-510S, using a spherical indenter) were also observed in the same way. Fig. 9 shows the cross-sectional SIM images of each indentation. Fig. 9(a) shows the cross-section of the impact indentation from LIPIT, revealing that nanoscale fine crystal grains were formed within the material near the indentation. On the other hand, Fig. 9(b) shows the cross-section of the indentation produced by the quasi-static indentation test. While a gradual change is observed in the SIM image, no fine crystal grains, as seen in the impact indentations from LIPIT, were formed. This difference in microstructure is believed to be due to variations in strain rate and strain magnitude. In particular, the strain rate of LIPIT reaches 10^7 s^{-1}, significantly higher than that of the quasi-static indentation test (10^2 s^{-1})[7].

Next, to investigate the improvement in mechanical properties due to the LIPIT impact indentations, nanoindentation tests were conducted near the impact indentations using the DUH-510S. The maximum test force and loading rate at this time were 10 mN and 1.49 mN/s, respectively, using a Berkovich indenter. Fig. 10 shows the results for pure iron. Fig. 10(a) shows a microscope image of the indentation points surrounding the impact indentation, Fig. 10(b) shows the indentation curve, and Fig. 10(c) shows the hardness distribution. These results indicate an increase in hardness near the LIPIT impact indentations, which was not observed in quasi-static loading indentations. It is believed that the microstructure of the pure iron substrate was made finer due to the high strain and high strain rate of plastic deformation, suggesting that LIPIT could serve as a new surface modification method.

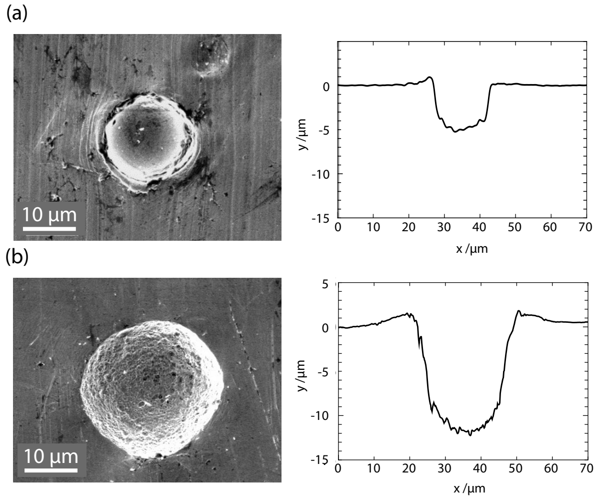

Fig. 8 SEM images and Cross-sectional Shapes of Impact Indentations on Pure Copper
(a) Impact Indentations from LIPIT with $15ZrO_2$
(b) Impact Indentations from LIPIT with $30ZrO_2$

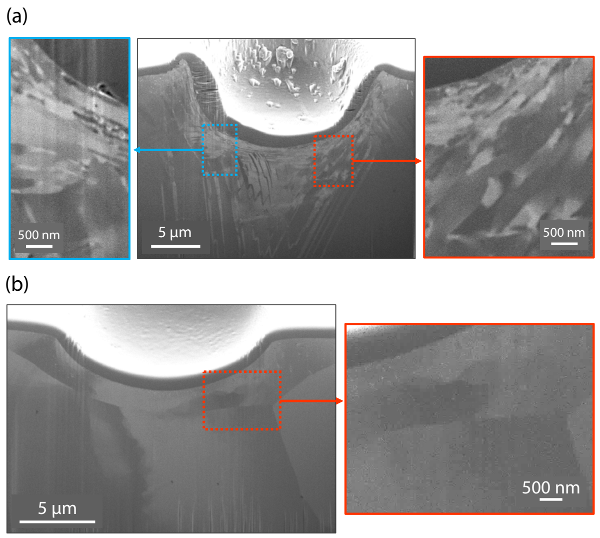

Fig. 9 SIM Images Obtained from the Cross-section of Impact Indentations on Pure Iron
(a) Impact Indentations from LIPIT with $15ZrO_2$
(b) Indentations from the Dynamic Ultra-Micro Hardness Tester

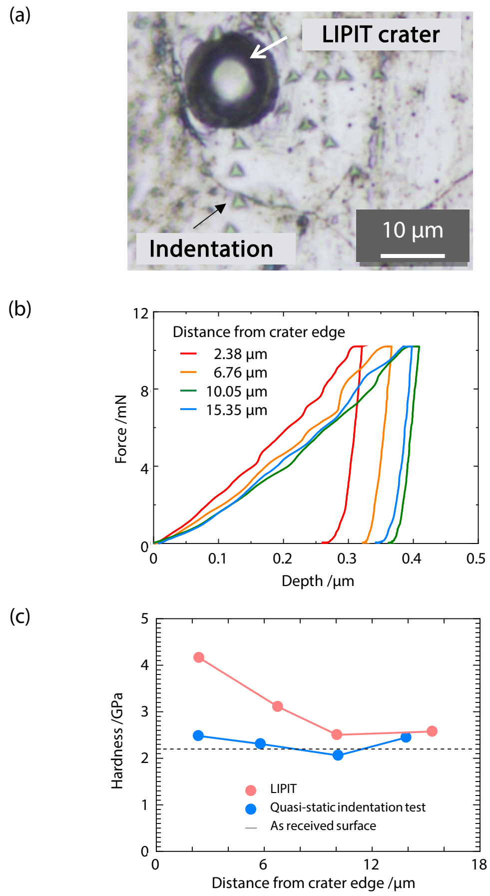

Fig. 10 Nanoindentation Tests around the Impact Indentation:
(a) Optical Microscope Image after Testing
(b) Indentation Curve at the LIPIT Impact Indentation
(c) Relationship between Distance from the Indentation Edge and Hardness

■ Conclusion

A LIPIT system was used to accelerate micro and nanoscale particles for high-velocity collisions, and a particle velocity measurement method was developed using mechanoluminescent materials. LIPIT is a unique technology for accelerating single or small quantities of microparticles for high-velocity collisions; however, measuring the flying velocity of the particles is crucial for further practical applications. Therefore, the flight behavior of the particles was captured using the HPV-X2 high-speed video camera, and the flight velocity was calculated from the recorded images. Additionally, an ML sensor using mechanoluminescent materials was developed to capture luminescence phenomena during particle collisions and measure the particle collision velocity. The usefulness of the ML sensor was validated by comparing these results with the measurements obtained from the high-speed video camera. Notably, luminescence was observed prior to the particle collisions with this sensor, and it was determined by visualizing it with the HPV-X2 that this was due to the collision of the scattered EA layer.

As described above, LIPIT technology enables collision testing of microparticles and makes it possible to create dynamic mechanical phenomena at the microscale and in nanosecond time. This enables the creation of new material properties and unknown mechanical phenomena, as well as having the potential to be developed as a surface processing technique. This article presents examples of surface modification methods for metal materials and suggests that the introduction of microstructures through high strain and high strain rates can lead to material strengthening. In the future, plans are in place to adjust particle velocity and shape to expand LIPIT into further surface modification technologies. To achieve this, visualization techniques for particle velocity and flight behavior are essential. The HPV-X2 introduced in this article is a high-speed video camera well-suited for visualizing the behavior of ultra-high-velocity particles, as demonstrated in this research.

Acknowledgments

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