High-Speed Infrared Imaging of Crack and Hot-Spot Formation in a Graphite-Fiber and Epoxy Composite Dynamic Load

The enormous progress made in the three last decades in high-speed thermal imaging have given rise to a wide variety of applications, of which material testing is maybe one of the most prominent. Split-Hopkinson bar experiments can especially benefit from the development of high-speed and high-resolution thermal infrared imaging. When materials are subjected to mechanical stress, rapid changes in the structure can occur. Being able to monitor hot spots prior to the breaking can help in the characterization of new material. In the present work, we have carried out high-speed infrared imaging at 20 000 frames per second (fps) during split-Hopkinson pressure bar experiments on graphite-fiber and epoxy composite materials to observe hot-spot formation and crack initiation. The results illustrate how high-speed infrared imaging can provide detailed information on the thermal properties of materials undergoing mechanical testing.

Introduction

The idea of using thermography for the characterization of material properties started in 1853 when Lord Kelvin found that elastically deformed bodies generate temperature changes when loading is applied. Tension causes cooling, while compression causes heating[1]. Later, in 1967, Belgen demonstrated that the thermoelastic effect could be used for stress analysis by applying infrared radiometry[2].

Infrared field temperature measurement techniques have therefore been used as a non-contact and real-time method for capturing the thermal energy being radiated from a surface in an inelastic deformation[3-5]. Jordan and Sandor, for example, have used differential temperature measurement for monitoring the elastic-plastic behavior of metals in 1978[6]. Following recent developments in thermographic techniques[7], quantitative stress measurements became possible by measuring the change in infrared photon emission produced by mechanical deformation.

Experimental mechanics testing could more than ever benefit from the improved performances of infrared cameras. Latest high-speed IR cameras provide high temporal and spatial resolution, which aid in the characterization of challenging targets. When a solid is submitted to dynamic loading, the energy conversion process leads to a concentration and conversion of diffuse mechanical energy in spatially confined regions, resulting in intense localized heating called hot spots. They are believed to be about 0.1 – 10 μm in diameter and may initiate chemical reactions in the material. Some of the hot spots theories have predicted relaxation times of 0.01 – 1 ms and temperatures of hundreds of Kelvin degrees[8]. A classic example of mechanochemical processes in solid materials related to the formation of hot spots is the mechanical initiation (by impact or shock) of explosions.

In the present work, we used the Telops FAST M3*k* combined with a microscope lens to investigate potential hot-spot formation and crack initiation in a composite material made of graphite fibers embedded in an epoxy matrix during split-Hopkinson bar experiments. Split-Hopkinson pressure bars were used to deliver loading pressure in the material in order to study the way loading stress induces cracks in the composite material, since crack initiation and growth can stimulate hot spot formation.

Experimental Set-Up

Telops FAST M3*k*

To be able to visualize thermal energy, an infrared camera with the capability of reaching high frame rates is required in order to localize energy at rates faster than thermal dissipation. The Telops FAST M3*k* used in this experiment (see Figure 1) is a cryogenically cooled high-performance infrared camera featuring a 320 × 256-pixel indium antimonide (InSb) focal plane array (FPA) detector covering the 3 – 5.4 µm spectral range. This model can reach a maximum speed of 3 000 fps at full frame and more than 100 000 fps in the sub-window mode.

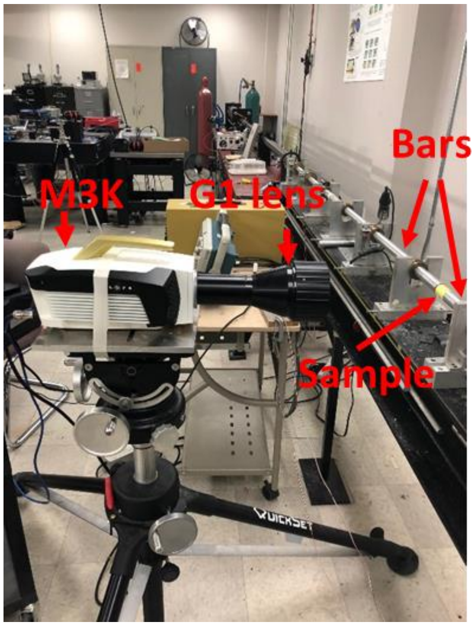

Figure 1 The split-Hopkinson pressure bar experimental setup with the Telops FAST M3*k* and the G1 1× lens.

A microscopic lens with 1× magnification, designed by Telops (G1 lens), was used for the experiments, leading to a spatial resolution of the order of the pixel pitch (30 µm). Another benefit of this lens is its long working distance of 25.8 cm, which helps to prevent risk of damage to the optic for applications where the sample might fragment. Sub-portions of 64 × 64 and 128 × 40 pixels were used with frame rates of 20 kHz (50 µs) and 19 kHz (52.6 µs) respectively.

Split-Hopkinson Pressure Bars

The split-Hopkinson pressure bar setup consists of a series of 3 bars. The sample is sandwiched between the incident and the transmitted bar (Fig.2). The striker bar is fired from a gas gun at a pressure of 5 psi and hits the transmitted bar, creating a stress wave. The stress wave then propagates through the sample. Part of the wave is reflected through the incident bar (reflected wave) while the remaining part propagates through the transmitted bar.

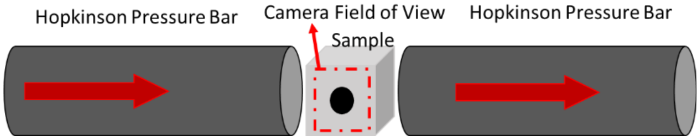

Figure 2 Illustration of the Hopkinson bars with the selected camera field of view.

The samples, made of graphite fibers embedded in an epoxy matrix, measured about 8 mm × 8 mm × 8 mm and contained various numbers of fiber inclusions. Three types of samples were studied: one with a single graphite fiber embedded in the epoxy matrix, one with two fibers close together, and one with two fibers a little farther apart. The graphite fibers were about 500 µm in diameter (see the 1000 µm blue scale in Fig.3 b and c), aligned parallel to the epoxy cube edges (Fig.3 b) or at a 45° angle (Fig.3 c). All samples were treated with a black coat layer before the tests to minimize the temperature variations induced by the emissivity difference between graphite and epoxy.

Samples

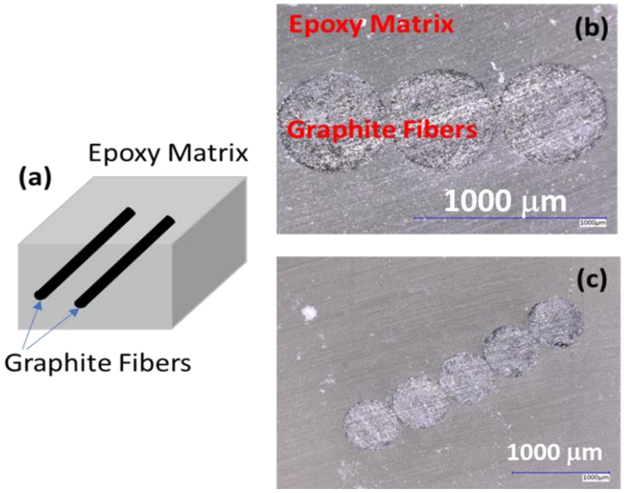

Figure 3 Illustration (a) and side view photographs (b,c) of the sample. Graphite fibers were embedded in an epoxy matrix.

For our experiments, we selected a camera field of view covering only the region of the sample containing the fiber inclusions.

Results and Discussion

Single fiber imbedded in epoxy matrix

For the test on a sample with one graphite fiber, a sub-window of 64 × 64 pixels and a frame rate of 20 kHz were used. Figure 4 shows selected infrared images recorded before, during and after the stress wave entered the sample. The images recorded before were used as a reference for image subtraction. Removing the background temperature allows to evaluate the net temperature increase during the experiment. When the sample is compressed by the bars, at the early stage one can see homogenous heat concentration around the graphite fiber with a temperature increase of about 2.5°C (frame labeled 0.15 ms). About 50 µs after the initial homogenous temperature increase, two hot spots (labeled A and B in Fig. 4) can be seen around the graphite fiber. These heat spots are most likely generated by tensile stress concentration on these points. Tensile strength in our epoxy matrix samples is indeed lower than the shear strength. Heat concentration and the opening-mode crack are therefore first expected around the tensile stress concentration points.

The first appearance of hot spots would also be expected around those points. In the three frames collected at a later stage (time labels 0.25, 0.3 and 0.35 ms), heat generation caused by the breaking of the graphite fiber is recorded. The separation between the epoxy matrix and the fiber is also clearly visible. About 0.1 ms after the observation of heat concentration generated by the tensile stress, two modes related to shear stress concentration around the fiber position can be seen. The heat shear modes appear more intense as the compression increases, and in the last three frames those shear modes reach maximum heat intensity. The observed dynamics appeared to be reproducible, as a total of four repeated trials were carried out on similar samples with similar results.

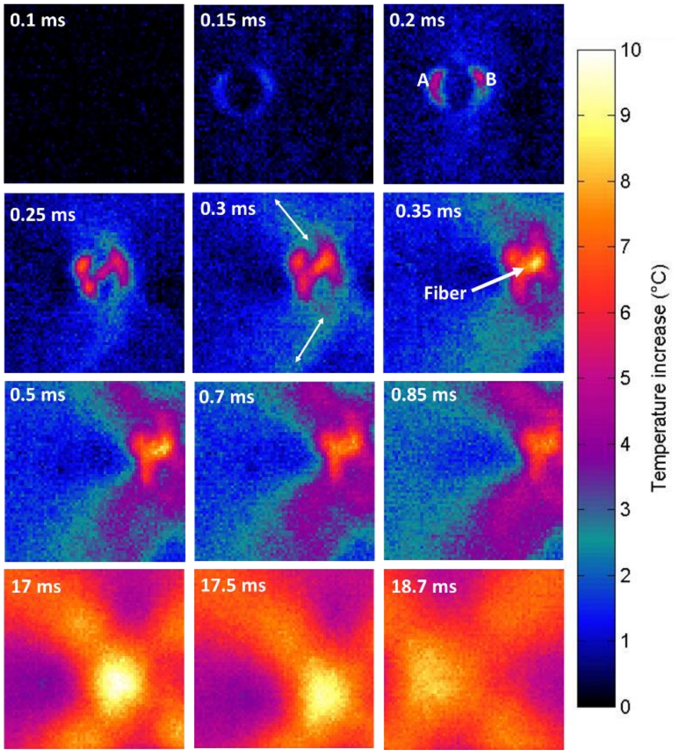

Figure 4 Selected infrared images of the split-Hopkinson experiment carried out on the sample with a single fiber.

Two fibers imbedded in epoxy matrix

In the two-fiber sample experiments, a sub-window of 128 × 40 pixels and a frame rate of 19 kHz were used. Two cases were studied: one with fibers almost in contact (Fig. 5 a), and one with a larger gap between the fibers (Fig. 5 b). The first case is referred to as "Geometry 1" and the other as "Geometry 2". The trial was performed on more than one sample of each geometry to confirm the reproducibility of the obtained results.

Photographs of the samples were taken before (Fig. 5 a, b) and after (Fig. 5 c, d) the test trial. Opening mode cracks parallel to the epoxy cube edges are visible in the images taken after the trial. The planar stress wave crossing over the graphite fibers and initiating the formation and growth of crack in the polymer material can stimulate hot spot formation in our samples.

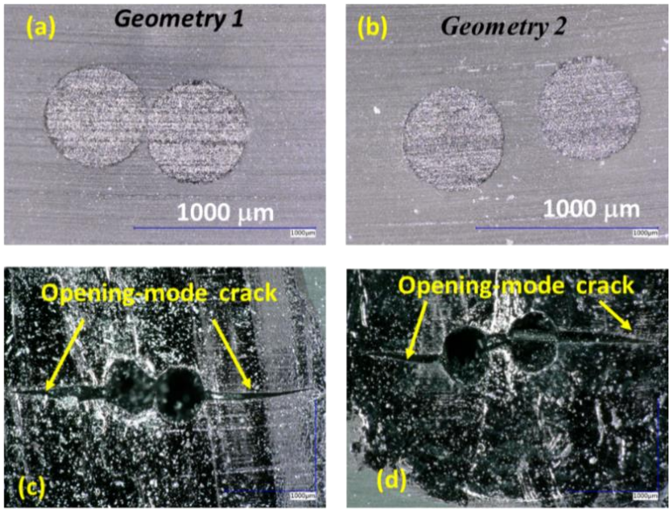

Figure 5 Photographs of the samples taken before (a, b) and after (c, d) the test trial.

In order to clearly visualize the heat exchange during the split-Hopkinson pressure bar experiments on the epoxy samples in Geometry 1 and 2, infrared images are shown frame by frame on Figures 6 and 7 respectively. When comparing the results of the two geometries, we can notice that the stress wave propagation in the material generates higher heat concentrations when the fibers are in contact. In Geometry 1, the tensile stress generates a temperature increase of about 2.5°C at about 0.16 ms (Fig. 6), while the temperature increase is negligible at the same timing in Geometry 2.

The fiber breaking behavior also appears to be different in the two geometries. We can see a clear breaking of the fibers at 0.21 ms in Geometry 2, while only the fiber on the right seems to break in Geometry 1 (Fig. 6 and 7). Heat concentration associated with frictions and/or tensile stress concentrations between the two fibers are also visible in Geometry 1, perpendicular to the broken fiber (frame labeled 0.21 ms).

In the following frames, we can observe that the fibers start to separate from the epoxy matrix. We can also note the development of heat modes, most presumably due to shear stress concentrations around the fiber. We noticed in all experiments that the fibers break about 0.1 ms after the stress wave enters the material, in a parallel direction to one of the shear stress modes' direction. This indicates that the shear stress is likely responsible for the breaking of the fiber, rather than tensile stress.

Finally, although tensile stress induced fractures and crack growths in the samples (Fig. 5 c, d), the temperature increase around the crack points was only of about 4.5°C. Hot-spot theories, however, predict temperature increases more than one order of magnitude higher than those measured in this work[8]. Further research on the matter would be needed; we suggest to conduct the same measurements on different polymer matrix composites, such as poly(methyl methacrylate) or polystyrene, to investigate the influence of their thermodynamic properties in the formation of hot spots. Loading pressure that causes opening crack modes due to both tensile and shear stress should also be used, since the shearing mode cracks also contribute to the hot spot formation process.

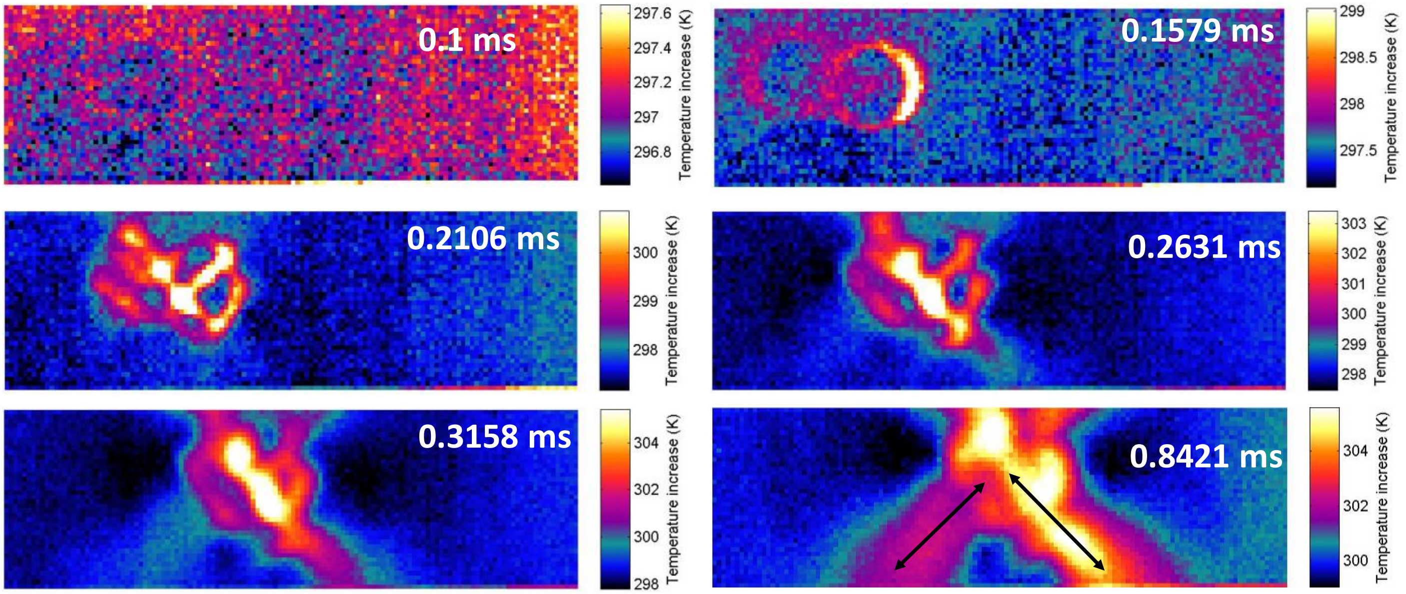

Figure 6 Selected infrared images of the split-Hopkinson experiment carried out on the sample shown in Fig.5a (Geometry 1).

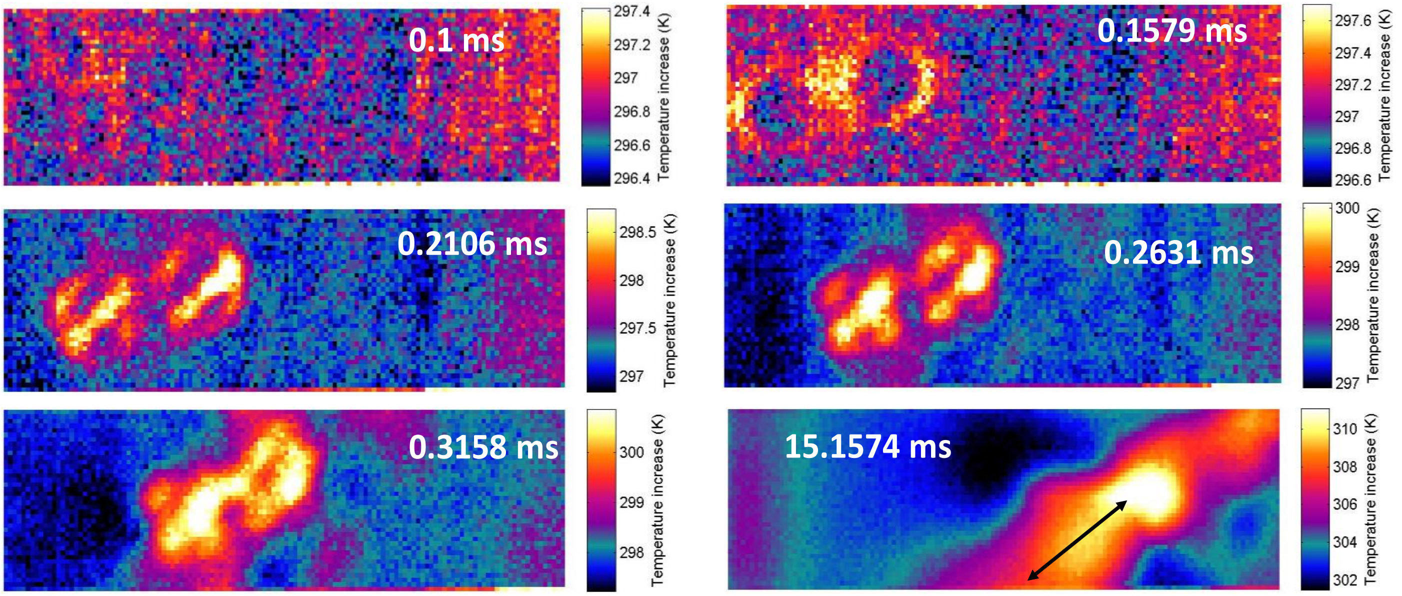

Figure 7 Selected infrared images of the split-Hopkinson experiment carried out on sample shown in Fig.5b (Geometry 2).

Fibers imbedded parallel and 45° in epoxy matrix

We studied the influence of the fiber orientation and density on heat generation dynamics caused by the stress wave propagation in the composite material. Figure 8 shows some photographs of the samples taken before the test trial. The fibers were aligned parallel (Fig. 8 a, c) and at a 45° angle (Fig. 8 b, d) to the epoxy cube edges. Only samples with fibers in contact with each other were considered this time, since this geometry has shown higher heat generation and more propagation dynamics (see above). We however varied the number of fibers in the matrix.

We used a sub-window of 128 × 40 pixels for the first three samples (Fig. 8 a, b, c) and of 128 × 64 pixels for the last sample (Fig. 8 d). The frame rates used were 19 kHz for the two first samples, 19 910 Hz for the third sample and 15 kHz for the last one.

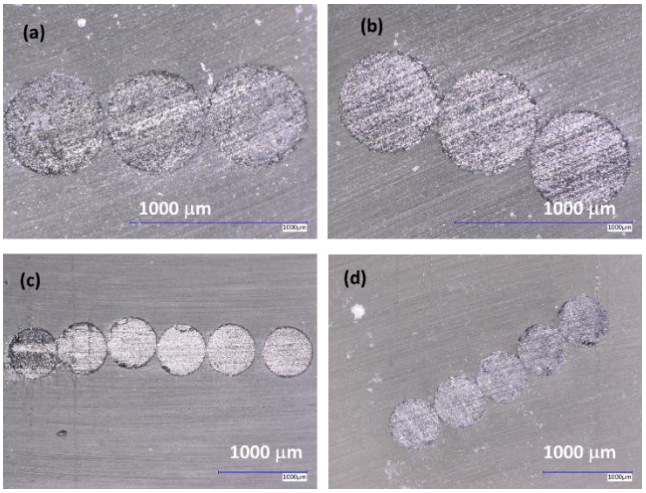

Figure 8 Photographs of the samples taken before the trials showing the fibers' alignment. They were either parallel (a, c) or at a 45° angle (b, d) to the epoxy cube edges.

Results are shown in figures 9, 10, 11 and 12. The dynamic observed on the first sample as shown in Fig. 9 is quite consistent with the results obtained with the two fibers in close contact (Geometry 1 in the section above). When the stress wave crosses the sample, the first and last fibers seem to be less damaged, while the breaking of the second fiber is very obvious (Fig. 9, frame labeled 0.1579 ms). The temperature rise is slightly higher in the three fiber configuration compared to the geometry with two fibers. Six hot spots of about 6°C were seen at an early stage in in the three fiber configuration.

When the fibers are aligned at a 45° angle (Fig. 10), the tensile stress seems to have a lower impact on heat generation. We observed a negligible temperature increase at a very early stage and the fiber breaking is less obvious compared to the sample shown in Figure 9. A total of three modes related to shear stress concentration can be seen in the geometry where the fibers are aligned parallel to the epoxy cube edges. One of these modes is parallel to the direction of second fiber breaking and the two other modes are parallel to the shearing strength direction that induces friction of the fibers. As for the 45° configuration, only one direction of the heat modes related to shear stress is seen. The influence of the fiber orientation in the observed dynamics is confirmed by the results obtained with the third sample, shown in figure 11 (consistent with the results of the first sample), and with the last sample, shown in figure 12 (consistent with the results of the second sample).

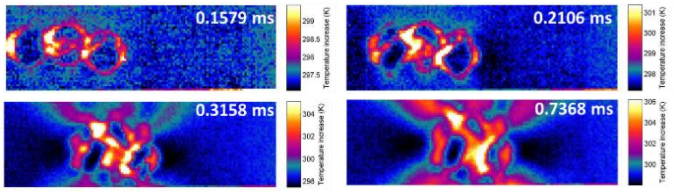

Figure 9 Selected infrared images of the split-Hopkinson bar experiment carried out on sample shown in Fig.8a.

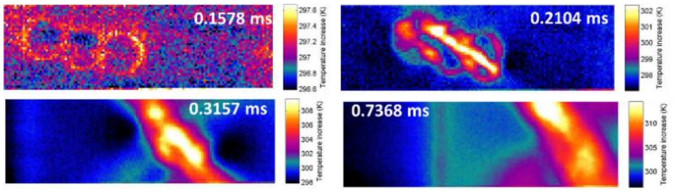

Figure 10: Selected infrared images of the split-Hopkinson bar experiment carried out on sample shown in Fig.8b.

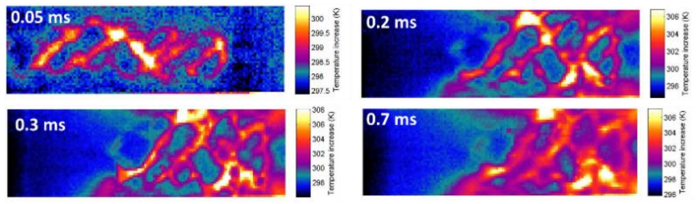

Figure 11: Selected infrared images of the split-Hopkinson bar experiment carried out on sample shown in Fig.8c

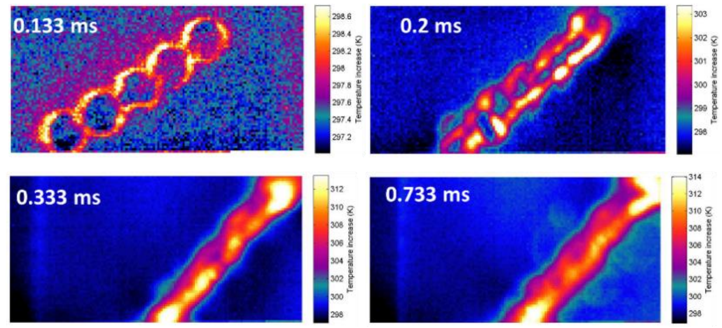

Figure 12 Selected infrared images of the split-Hopkinson bar experiment carried out on sample shown in Fig.8d.

Conclusion

We have investigated heat generation dynamics in an epoxy/fiber composite material. With the Telops' FAST M3*k* camera and the G1 1X microscope lens, we were able to reach a temporal resolution of 50 µs and a spatial resolution of a few tens of mm, which was ideal to investigate crack initiation and hot-spot formation within the materials.

We were able to observe the effect of both tensile and shear stress on the fibers. The breaking was mostly due to shear stress. Some moderate tensile-stress-induced hot spots were measured, but further research is needed to understand the influence of the thermodynamic properties of the material in the formation of hot spots. However, we observed a clear influence of the fiber alignment and density in the epoxy matrix on heat generation and the breaking dynamics of the fibers.

Acknowledgements

Telops would like to acknowledge the support of Prof. Addis Kidane and Dr. Suraj Ravindran of the Dynamic Behavior and Multifunctional Materials Laboratory (DBMML) of the University of South Carolina,Columbia, USA, in this work.

References

[1] Thomson, IF. (Lord Kelvin), "On the Dynamical Theory of Heat", Trans. Roy. Sac., **20**, 261-283, 1853.

[2] Belgen, M.H., "Infrared Radiometric Stress Instrumentation Application Range Study", NASA Report CR-1067, 1967.

[3] Reifsnider, K.L. and Williams, R.S., "Determination of Fatigue related Heat Emission in Composite Materials", Experimental Mechanics, **14**(12), 479-485, 1974.

[4] Marcus, L.A. and Stinchcomb, W., "Measurement of Fatigue Damage in Composite Materials", EXPERIMENTAL MECHANICS, **15**(2), 55-60, 1975.

[5] Charles, J.A., Appl, F.J. and Francis, I.E., "Using the Scanning Infrared Camera in Experimental Fatigue Studies", EXPERIMENTAL MECHANICS, **15**(4), 133-138, 1975.

[6] Jordan, E.H. and Sandor, B.I., "Stress Analysis from Temperature Data", J. Test. and Eval., **6**(6), 325-331, 1978.

[7] Mountain, D.S. and Webber, J.M.B., "Stress Pattern Analysis by Thermal Emission (SPATE)", Proc. Sac. Photo-Opt. Inst. Engrs., **164**, 189-196, 1978.

[8] F. P. Bowden, A. D. Yoffe, and G. E. Hudson, Initiation and Growth of Explosion in Liquids and Solids, Cambridge University Press, 1952.

[9] Bowden, F. P. & Yoffe, A. D. Initiation and Growth of Explosion in Liquids and Solids, Cambridge University Press, 1952.

[10] Langer, G. & Eisenreich, N. Hot spots in energetic materials. Propell. Explos. Pyrot. **24**, 113–118, 1999.

[11] Field, J. E. Hot spot ignition mechanisms for explosives. Acc. Chem. Res. **25**, 489–496, 1992.

[12] Armstrong, R. W., Ammon, H. L., Elban, W. L. & Tsai, D. H. Investigation of hot spot characteristics in energetic crystals. Thermochim. Acta **384**, 303–313, 2002.

[13] Dlott, D. D., Peter, P. & Jane, S. M. in Theoretical and Computational Chemistry Vol. 13, Ch. 6, 125–191, 2003.

[14] J. E. Field, Acc. Chem. Res. **25**(11), 489, 1992.

Telops Inc.
100-2600 St-Jean Baptiste Ave
Québec, QC, Canada, G2E 6J5

+1-418-864-7808
sales@telops.com
www.telops.com