Thermal expansion of graphite intercalation compounds

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Graphite and various composite materials based on graphite, including graphite intercalation compounds (*GICs*), are widely used in a variety of fields of science, technology, and industry [1]. Some *GICs* with acids and salts are used to obtain thermally expanded graphite (TEG), which has the form of a foamed carbon structure and is used to produce low-density carbon materials[2-4].
Expansion of *GICs* consists in separating the individual layers in a more or less regular manner, such a separation being sufficient to remove all the inter-planar interaction. For that purpose, the presence of an intercalate is necessary: heating the *GICs* induces the vaporization of the intercalated species and hence a significant expansion of the material along the crystallographic c-axis occurs [1-4]. Thermal expansion mechanism of *GICs* is investigated by using infrared (IR) laser irradiation.
The specifically designed optical set up allows for measuring the critical temperature which must be exceeded for exfoliation of intercalated graphite flakes (provided by Asbury Carbons) and transition to expanded filaments of graphite flake. An infrared laser at wavelength 1.07 µm was employed to induce a thermal shock and the temperature increase was measured by detecting in the 8-12 µm the mid-infrared radiation emitted by a single grain during the heating process. The laser head was positioned in a vertical plane and the laser beam impinges at incident angle 45° on the working surface mounted on an X-Y translation stage (fig. 1 right). The thermal imaging system consisted of a pyro-electric detector array (Pyrocam III by Spiricon Ophir) composed by 124x124 pixels with 85 µm pitch and 100 µm element spacing. This detector is internally chopped and thermal images are recorded by a computer up to a maximum speed of 48 frames/s. The infrared radiation emitted by a grain of graphite flakes was collected and imaged over the detector by means of a germanium close-up lens system, which acted also as a filter for any radiation out of the 8-12 µm range. The whole IR detecting apparatus was mounted on the working surface where the grain was located and it allowed to record 1:1 thermal images of the graphite grains during the laser irradiation process (Fig. 1-left).

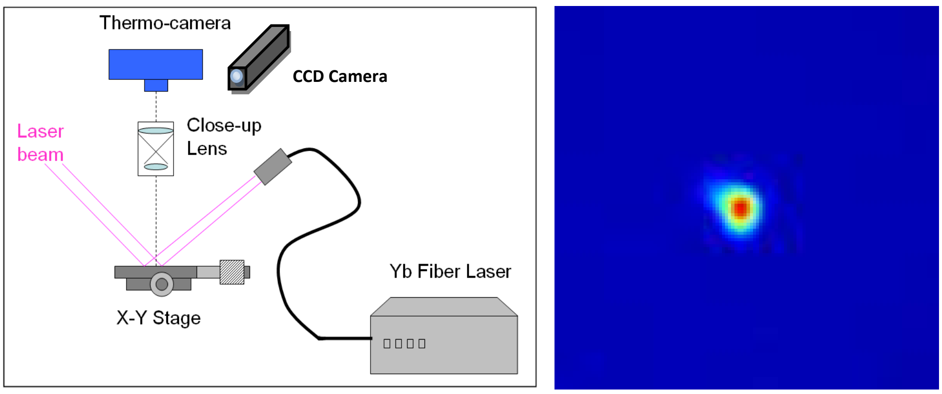

Fig. 1: Experimental setup and thermal image of a graphite grain.

Thermal data have been obtained from sequence of recorded IR frames. Fig. (2a) shows a typical three dimensional reconstruction of temperature field form a thermal image of a graphite flake. Fig. (2b) shows the typical temporal evolution of the IR emission from the grain central position during the laser irradiation process. The abrupt increase of the temperature corresponds to a critical transition temperature of the grain of about 160°C.

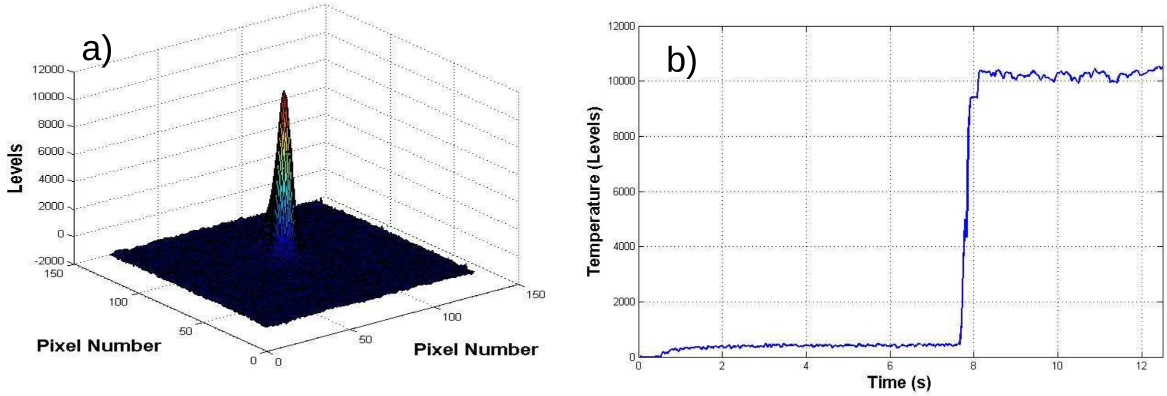

Fig. 2 Laser induced graphite grain temperature; (a) three-dimensional reconstruction of the spatial distribution of the temperature (Pixel Spacing 100μm);(b) typical temporal evolution of the IR emission from the grain central position during the laser irradiation process

The real time temporal evolution of the expansion process is recorded by an high speed (250 frame/s) video camera in the visible range, mounted in the vertical plane orthogonal to the laser beam direction. This camera was equipped with a macro lens in order to obtain greatly detailed images of the grain (37 pixels/mm) and to follow directly the temporal evolution of the laser induced thermal expansion process Fig. (3a) shows the sequence of video frames recorded by the visible camera. These sequence clearly shows the extrusion of graphite filament. The grain increases about 100 times its initial volume and the process is accompanied by vaporization of the intercalated species.

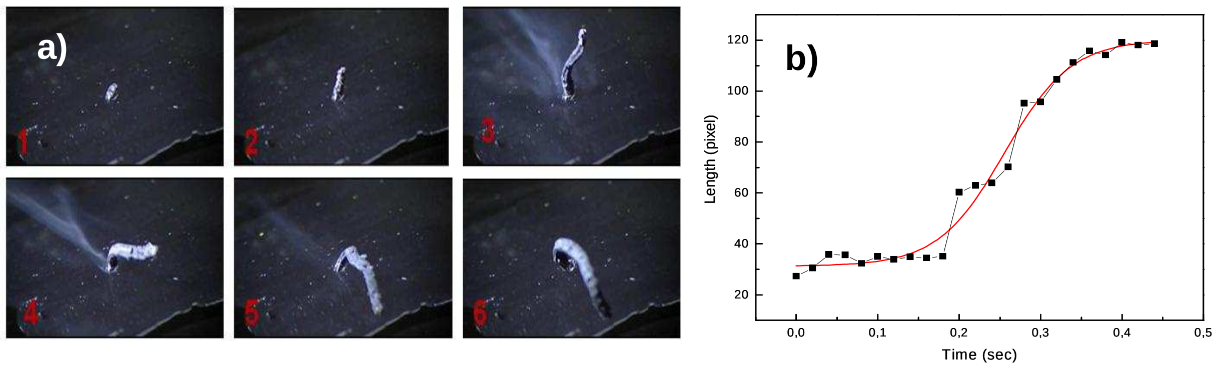

Fig. 3: Temporal evolution of the grain transition.

The length of extruded flake is obtained by image processing (Sigma Scan Pro 5) the recorded video frames and its temporal evolution is shown as a function of the irradiation time in fig. (3b).
In conclusion we investigated the laser induced thermal exfoliation of graphite intercalation compounds A flexible optical set-up was developed for real time monitoring the extrusion process of the graphite flakes in the visible and the temperature field distribution across the grain size during the laser irradiation process. Temperature measurements performed over different sized grain indicated a critical transition temperature less than 190°. The extrusion process were found to last few seconds depending on the laser irradiation power.

References

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