A NOVEL METHOD ON VISUALIZATION OF TEMPERATURE FIELDS BY PYROLYTRIC GRAPHITE SENSORS AND IR DETECTION SYSTEMS

Maciej Miszczak[1], Waldemar Świderski[1]

[1] Military Institute of Armament Technology
Wyszynskiego 7 Str., 05-220 Zielonka, Poland

Corresponding author: Waldemar Swiderski, waldemar.swiderski@wp.pl

Abstract: A novel, experimental method on nonintrusive visualization of temperature fields caused by immobile and moving media – solids and liquids of temperature higher than ambient one - with use of pyrolytic graphite (pyrographite) sensor and IR thermal camera, is presented. This visualization was achieved thanks to the direct contact of internal pyrographite surface/wall of high heat transfer coefficient with given medium and due to usage of IR thermal camera watching external pyrographite surface/wall opposite to the internal one. In addition, in the case of moving medium, adjustment (orientation) of pyrographite parallel, crystallographic basal planes across, preferably perpendicular to the direction of medium movement, makes possible visualization of medium displacement. Such measurements can be applied in the wide range of media temperatures, i.e. from relatively low (e.g. 30^{o}C) to very high ones (e.g. 3500^{o}C caused by combustion process) because of pyrographite exceptional high anisotropy of heat conductivity. Another pyrographite feature making it very suitable in IR detection, it is very high thermal emissivity (0.95-0.97).
Key words: pyrolytic graphite, IR detection, temperature field, solid and liquid medium

1. INTRODUCTION

Common measurements of field temperatures and heat fluxes caused by various media, are mainly based on application of thermocouples or modern IR detecting systems like thermal cameras (NATO RTO Educational Notes - RTO-EN-AVT-117, 2004). The latter ones became the most powerful tools for non-intrusive and enough precise determinations of temperature fields. Well known limitations of thermocouples usage result from fact that they introduce certain disturbances in temperatures measurements by their presence, and in majority they are relatively susceptible for degradation under severe conditions, i.e. at high temperature and/or under influence of aggressive chemical environment (FR/GE/UK/US International Test Operations Procedure No. 5-2-500, 2000). On the basis of cross-reference analysis of scientific literature incl. patent documents, the closest solution to the idea presented in this article, it seems to be an application of pyrographite gauge for measurements of heat fluxes (Bunker et al., 2002), consisted of pyrographite slug with its heat flux receiving surface of high heat transfer coefficient and at least one thermocouple embedded in the slug. Such design of the pyrographite gauge, allows to use thermocouples successfully for heat fluxes measurements under very severe conditions e.g. created by high temperature and destructive chemically and mechanically high-speed flows of combustion products in combustion chambers and in the nozzle ducts of rocket motors during propellant charges burning. Exceptionally high thermal, mechanical and chemical resistance of pyrolytic graphite against erosion by combustion products, is commonly utilized in rocket propulsion systems, especially in their nozzle assemblies, in which pyrographite is used as insert of nozzle in its throat section (of critical diameter), i.e. where there are the most severe impact of combustion products flow (Sutton, Biblarz, 2001, Miszczak et al, 2007, Miszczak, Świderski, 2008, Florczak et al., 2012).

An idea of usage of the pyrolytic graphite in quite new area of science and technology, i.e. as thermal sensor element together with IR thermal camera for detection of temperature fields, emerged during tests on combustion process (Świderski et al., 2008; Świderski, Miszczak, 2009; Miszczak et al., 2009; Świderski et al., 2011) of homogeneous (double-base) rocket propellants. According to these tests, rocket propellants in the form of end burning charges (cylindrical strands) were inserted into the pyrgraphite tubes and ignited by CO_2 laser from one end. Their burning rates under these conditions were in the range 1-2 mm/s. The pyrographite tubes, except playing role as thermoresistant, construction elements housing tested high energetic charges, first of all performed function as thermal management elements guiding through their walls in specific way heat fluxes generated by combustion zones during burning of these high-energetic charges. Such guidance of thermal fluxes was caused by unique thermal conductivity anisotropy of pyrographite resulting in high thermal conductivity along the pyrographite tube radius and low thermal conductivity along the pyrographite tube axis. Due to this anisotropy and direct contact of the pyrographite tube inner surface with cylindrical surface of tested high energetic charge, it was possible visualization and registration of combustion zone movement by IR thermal camera, as a heat zone traveling from ignition point, axially, along external, side surface of the pyrographite tube. From sequence of IR thermal images, histories of temperature fields (profiles) along external, side surface of the pyrographite tubes were obtained. On the basis of quasi-continuous measurements of changes of positions of arbitrary selected isotherms of the heat zones during combustion process, burning rates of tested propellant samples were determined.

Observations obtained from above investigations on combustion zones propagations directed our attention to check possibility to visualize movement of media of temperature below their ignition temperatures and towards measurements of temperature fields caused by such moving or immobile media. This new idea seems to be very promising in application of pyrographite sensor-IR thermal camera visualization systems in industry and technology areas as monitoring, controlling and surveillance systems, especially in thermal, nuclear engineering, chemical technologies, and metallurgy (metal) processing.

2. EXPERIMENTAL

2.1 Tested materials, experimental set-ups and measurement procedures

The following tested media were used. As solid material samples – copper, aluminum, steel, brass and glass in the form of cylindrical rods were applied. Fluids used in the tests, were liquids – oil and distilled water.

The tested, solid samples heated to the constant temperature in the range 20-400^{o}C by external electro-heating source, were partly inserted into the pyrographite tubes keeping direct contact through their side surfaces (Fig.1, 2). Optionally the rods were immobile in the pyrographite tube (Fig.1) or they were moved in its axial direction (Fig.2). The tubes were consisted of pyrographite rings of internal and external diameter of 5.0 mm and 10.0 mm respectively. Each pyrographite ring had height between 5.0 mm and 6.0 mm. The pyrographite rings were bonded to each other through their head surfaces by thermoresistant adhesive. The total lengths of the pyrographite tubes were in the range 15.0-30.0 mm.

The tested liquids heated to the determined temperature between 40-60^{o}C, were injected into the narrow conduit of diameter of ca. 1 mm and length of 40 mm, drilled through the pyrographite stack of total height of 40 mm made of pyrographite rings of external diameter 35 mm and internal diameter 15 mm, bonded through their head surfaces (Fig.3). The height of each pyrographite ring in the stack, was in the range 4.0-6.0 mm. This narrow conduit was parallel to longitudinal axis of the pyrographite stack, and was located at the distance of 3.0 mm from the external, side surface of the pyrographite ring stack. Such location of the conduit resulted in its eccentric position in relation to the longitudinal axis of the pyrographite stack.

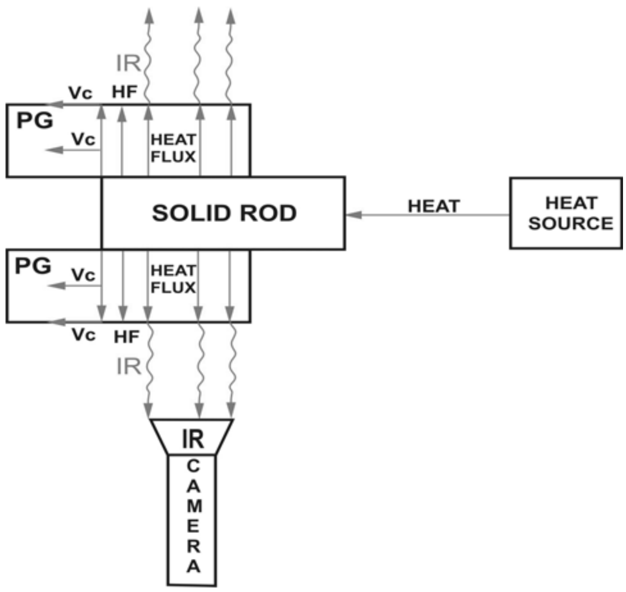

Fig.1. Schematic diagram of experimental set-up measuring by IR camera temperature field evolution on the external, side surface of the pyrographite (PG) tube, induced by the immobile solid rod inserted into the PG tube and heated by external heat source; HF-heat front (directly generated by hot immobile solid rod) moving in the PG tube wall with V_C velocity along the PG tube axis.

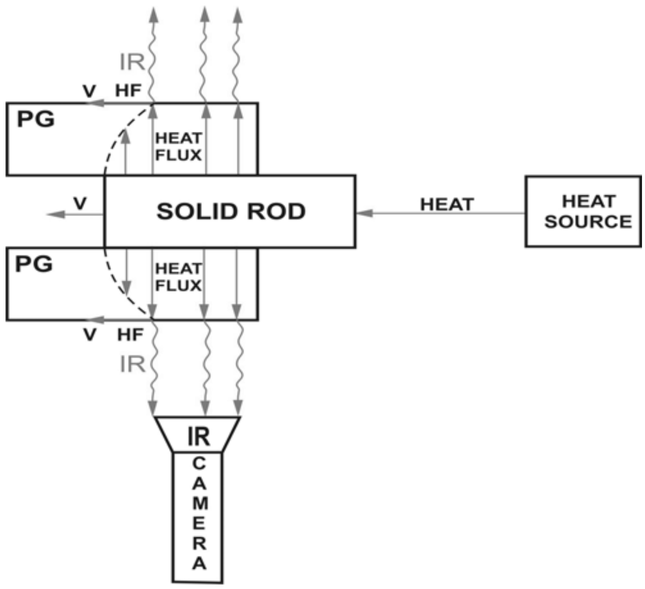

Fig.2. Schematic diagram of experimental set-up measuring by IR camera temperature field evolution on the external, side surface of the pyrographite (PG) tube, induced by moving with V velocity solid rod inserted into the PG tube and heated by external heat source; HF-heat front moving with V velocity on the external, side surface of the PG tube, along its axis.

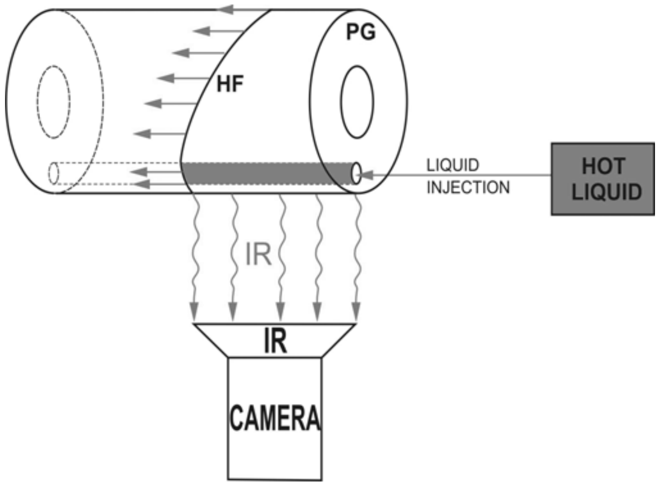

Fig.3. Schematic diagram of experimental set-up measuring by IR camera temperature field evolution on the external, side surface of the pyrographite (PG) stack of rings, induced by movement of hot liquid flowing through the open in both ends, narrow, eccentric conduit made in the PG stack, parallel to its longitudinal axis; HF- Curved heat front/zone moving on the external, side surface of pyrolytic graphite stack with the same velocity as head of hot liquid stream in the eccentric conduit.

The pyrolytic graphite used as material for rings of tubes and stacks made from rings, was manufactured in the Military Institute of Armament Technology (Zielonka, POLAND) by means of chemical vapor deposition (CVD) of hydrocarbon gas at low pressure passing over the appropriate substrate held at a high temperature in the chamber of special, experimental furnace equipped with inductive heating system. Due to above method and apparatus for manufacture of pyrolytic graphite, substrate – six plate matrices made from isotropic graphite, were mounted in the furnace chamber forming duct of hexagonal cross section. As hydrocarbon gas – pure propane under pressure of 8 hPa was fed to the duct through the inlet made in the upper cover of the furnace. The temperature of the duct walls, was kept in the range between 2080^{o}C and 2120^{o}C Propane flowing through the duct was subjected to pyrolysis resulted in deposition of the pyrolytic graphite on the substrate plates and in release of post-pyrolysis (post-pyrolytic) gas which was directed to the outlet of the bottom cover of the furnace. After completion of the pyrolysis process, the upper and the bottom covers of the furnace were cut off and the substrate plates with deposited layer of the pyrolytic graphite were separated each other. Then, the layers of deposited pyrolytic graphite were carefully removed from the substrate plates and finally the pyrographite layers - plates were machined and finished to the form of the rings. Each pyrolytic graphite ring had mass density of 2.19 g/cm^3, and the distance between its adjacent crystallographic parallel, basal planes, i.e. layer plane spacing, was 0.342 nm. Thermal conductivity of the pyrolytic graphite was 350 W/(m·K) and 1.77 W/(m·K) respectively along its parallel, basal planes and in direction perpendicular to them. The mass density of pyrolytic graphite was determined by gas pycnometer. Such type of pycnometer is suitable for pyrographite density determination because of its very small (residual) porosity and gas permeability. The distance between adjacent basal planes of the pyrographite was determined using DRON 1 diffractometer in a monochromatic Cu Kα radiation. X-ray diffraction (XRD) patterns contained reflexes belonging to the (002) and (004) pyrographite crystallographic planes. The thermal conductivity coefficient of pyrolytic graphite along its basal planes and perpendicular to them, was determined by method of indirect calculations using measured values of pyrographite thermal diffusivity, thermal capacity at constant pressure and mass density. The thermal diffusivity was determined by thermal pulse method using laser, the heat capacity at constant pressure was determined by differential scanning microcalorimeter (DSC). These pyrographite parameters, especially its thermal anisotropy and structure, gave basis for assumption that each pyrographite ring forms macro-crystal composed of parallel, basal crystallographic planes. Such crystallographic structure results in high thermal conductivity along radius of the pyrographite ring (tube, stack) and low thermal conductivity along its height. In this place it is worth to mention that critical importance in maintenance of parallelism of pyrographite basal planes in macro-scale had smoothness of substrate surface on which pyrolytic graphite was deposited, purity of hydrocarbon gas and its flow parameters including flow velocity, pressure and temperature during pyrolysis process. Above assumption on parallelism of pyrographite basal planes in the all volumes of the pyrographite rings and as consequence - in all volume of the pyrographite tubes or pyrographite stack consisted of rings, was additionally confirmed by means of IR thermal visualizations.

IR thermal detections were realized by means of thermal camera FLIR SC 7600 capable to register with high resolution at speed up to 300 fps. The thermal IR camera watching external, side surface of the pyrographite tubes and pyrographite stack, was used for detection and registration of temperature fields caused by solid and liquid media.

2.2 Test results and their discussion

Exemplary, representative IR thermal images of temperature fields on the external, side surface of the pyrographite tubes-sensors obtained by means of IR camera operated with speed of 7 fps, are presented in Fig. 4, 5,6 for immobile solid medium - copper rod of temperature 50^{o}C inserted in the pyrographite tube and - in Fig. 7, 8 - for hot oil of the temperature ca. 60^{o}C flowing through the narrow, eccentric conduit made in the pyrographite stack.

To Fig.4, 5, it corresponds Fig.6 which describes temperature field evolution obtained on the basis of IR measurements on the external, side surface of the pyrographite tube with partly inserted immobile hot rod.

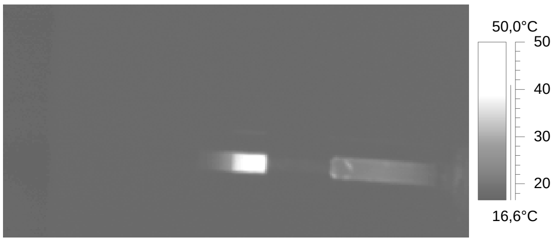

Fig.4. IR image of temperature field on the external, side surface of the pyrographite (PG) tube after its heating for 60.38 seconds by immobile copper rod of temp. ca. 50^{o}C partly inserted into the PG tube.

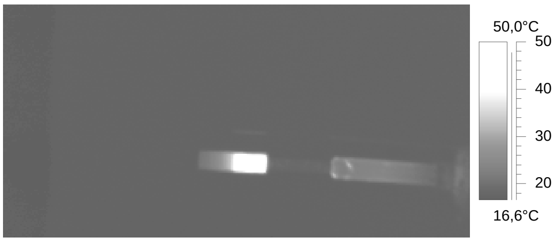

Fig.5. IR image of temperature field on the external, side surface of the pyrographite (PG) tube after its heating for 124.88 seconds by immobile copper rod of temp. ca. 50^{o}C partly inserted into the PG tube.

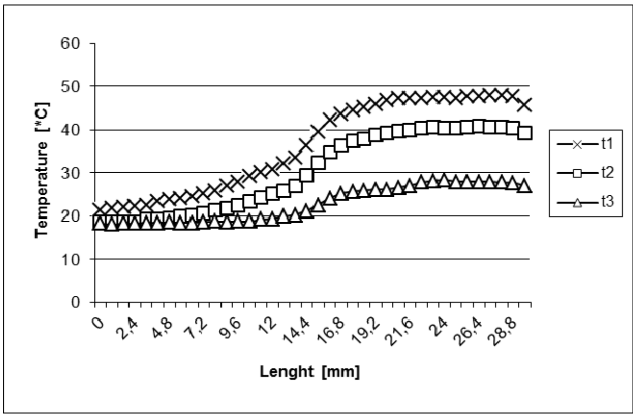

Fig.6. Measured by IR camera evolution of temperature distribution along external, side surface of the pyrographite (PG) tube after its heating for t1 = 124.88 s, t2 = 60.38 s, t3 = 25.35 s periods by immobile copper rod of temp. ca. 50^{o}C partly inserted into the PG tube.

In the case of immobile copper rod usage, IR thermal images (Fig.4,5) and Fig.6 show distinctly visible and sharp enough heat zones emerging and moving relatively slow on the external, side surface of the pyrgraphite sensor tube along its axis. These heat zones consisted of isotherms, were circular, so they were registered by IR thermal camera as vertical ones to the tube axis (Fig.4,5). It indicates that heat fluxes from solids had the same distance to achieve external, side surface of the pyrographite tube. The heat zones (their isotherms) slowly moved along axis of the pyrographite tube (Fig.4,5,6) due to relatively low thermal conductivity of the pyrographite in direction vertical to its crystallographic basal planes. The movement of heat zones along the axis of the pyrographite tube was additionally delayed from the reason of presence of low thermal conductivity adhesive used for bonding the pyrographite rings.

In the case of flows of liquids (their movement) along the narrow, eccentric conduit (Fig.3), heat zones were not observed as vertical ones as in the case of the solid rods. These zones had curved contour (Fig.3,7, 8) which resulted from eccentric location of narrow conduit made in pyrographite ring stack.

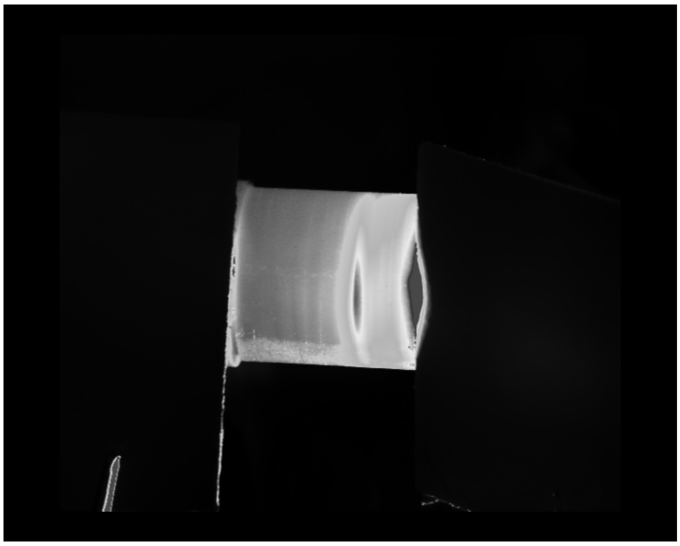

Fig.7. IR image of curved heat front moving on the external, side surface of pyrographite (PG) stack at the beginning of hot liquid flow through the open (from both ends) eccentric, narrow conduit drilled in the (PG) stack.

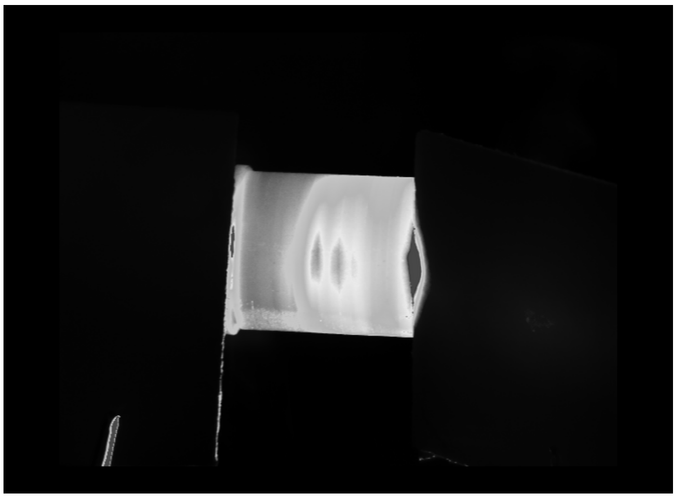

Fig.8. IR image of curved heat front moving on the external side surface of pyrographite (PG) stack during later stage of hot liquid flow through the open (from both ends) eccentric, narrow conduit drilled in the (PG) stack.

The shapes of above curved contours were also dependent on positioning of IR camera in relation to the eccentric channel and - on velocity of flowing liquid. With increase of flow velocity of the liquid in the eccentric conduit, the curvature of the contours of external isotherms increases as well. In Fig. 7, 8 the distance between the IR camera and the narrow channel was the shortest, so the moving curved envelope of the external heat zones were symmetrical convex in direction of their propagation. The most forward (top) moving point of curved heat zones was generated by heat fluxes which had the shortest distance to achieve external, side surface of the pyrographite stack. With increase of distances between liquid stream (conduit) and the side surface of the pyrographite stack, the distances of heat fluxes to achieve external side surface of the tube, increase resulting in higher delay time of appearance of points forming curved heat zone on the external surface. Thus, these points are situated more behind the most forward (top) point of the heat zone. In addition, it was also observed by IR thermal camera, that curved contours of external heat zones started to grow (spread) simultaneously (symmetrically) from the most forward (top) moving point to both sides situated behind this point. This visual phenomena was resulted from the differences in distances to travel by the heat generated from head/front of moving liquid in the eccentric conduit to external surface of the pyrographite stack/tube.

When the liquid velocity decreases and finally it stops in the eccentric conduit, the curvature of external heating zones (external isotherms) decreases (it "flattens") and then it becomes straight (vertical) moving slowly along the axis of the conduit (stack).

In the case of moving media, measuring displacements of the isotherms visualized by IR thermal camera on the external, side surface of pyrographite sensor-tubes/stacks and measuring time needed for such displacements on the basis of periods obtained from the sequence of IR thermal images, it is possible to determine quasi-continuous or average velocity of given medium front/zone movement/displacement.

3. CONCLUSIONS

On the basis of presented experimental measurements and observations, it is possible to withdraw the following conclusions:

1)Visualization of temperature fields caused by immobile and moving media – solids and liquids - with use of pyrolytic graphite (pyrographite) sensor tube/stack and IR thermal camera, is possible if the following circumstances are fulfilled:

- it is ensured direct contact of tested medium material of temperature higher than ambient one with internal, side surface of pyrographite tube, i.e. with surface of high heat transfer coefficient;
- it is applied IR thermal camera watching external, surface of pyrographite sensor, i.e. surface of high heat transfer coefficient.

2) Visualization of movement of solid and liquid media of temperature higher than ambient one, is possible if the above requirements are met, and additionally, when pyrographite parallel, crystallographic basal planes of the tube/stack-sensor are directed across, preferably perpendicular to the vector (direction) of medium propagation and when the velocity of medium movement is higher than the velocity of heat zone induced by immobile medium, traveled through the wall of the pyrographite tube/stack in direction perpendicular to its basal crystallographic planes. Using signs/symbols used in Fig.1,2, the latter requirement can be expressed as: $V > V_c$. The value V_c was roughly estimated as ca. 0.25 mm/s. This limit value V_c highly dependant on thermal conductivity of pyrographite in direction perpendicular to its basal crystallographic planes, was estimated on the basis of measurement of time necessary to travel distance of ca. 5.0 mm on the external, side surface of the pyrographite tube, i.e. along the height of one pyrographite ring, by arbitrary selected isotherm generated on this surface by immobile solid rod (Fig. 1,4,5,6).

3) In the case of moving medium (solid rod, liquid), position of heat zone generated on the external surfaces of pyrographite tube/stack by such medium, corresponds to the location of front/head zones of this medium. Between position of front/head zone of moving medium generating heat towards external surface of pyrographite and position of heat (isotherm) on the external surface of pyrographite sensor, it exists certain spatial/time shift dependant on the distance to travel by heat between these positions. Above spatial/time shift dependence is distinctly illustrated as curved isotherms on the external surface of pyrographite ring tube/stack (Fig.3,7,8).

4) Because of exceptional high thermal resistance and unique thermo-physical properties esp. thermal conductivity anisotropy of pyrographite, and also due to its high IR radiation emissivity in combination with detection capabilities of IR thermal camera characterized by its high sensitivity and resolution to time-distance registrations and temperature distributions, supported by efficient digital processing software of IR images, presented experimental set-up configurations allow to measure wide range of temperature fields caused by tested media, from low temperatures, i.e. temperatures of some degrees higher than ambient temperature (e.g. 30^oC) to very high temperatures (e.g. 3500 ^oC) caused by combustion processes.

5) Pyrographite, due to its high thermal conductivity along basal crystallographic planes (comparable with thermal conductivity of copper) and due to relatively high heat capacity, indicates high cooling ability

which is well illustrated in Fig.6, where it was needed relatively long time - grater than two minutes to achieve temperature close to 50^oC on the external surface of pyrographite tube, generated by the immobile copper rod of temperature 50^oC inserted in the tube.

6) Pyrographite is suitable not only for IR thermal visualizations but it could be also very useful for VIS temperature mapping and detection of heat zones movement as "black body" substrate (which does not introduce own clutters in VIS region) for thermochromic thin layers (films), especially - for thermochromic liquid crystals like chiral-nematic (cholesteric) ones.

7)The pyrographite sensors can be applied as sensitive thermally "windows" for nonintrusive measurements of temperature fields caused by immobile or moving media monitored and surveyed by distant, remote IR detection systems. Such windows can be embedded in the walls made of other types of materials. The pyrographite windows can be mounted in the walls of any ducts, conduits transporting warm or hot solids, fluids (the latter ones being under relatively high pressure). Such pyrographite windows can be also embedded in the walls of laboratory and industrial ovens incl. furnaces, and also in reactors applied e.g. in chemical technology. The pyrographite windows embedded in the walls should be thermally isolated from the wall materials, both in order to avoid too rapid and intensive thermal influence of wall materials adjacent to the window, which can disturb thermal response of the pyrographite window to temperature changes caused by immobile (stationary) or moving (flowing) media.

8) Above presented novel method on nonitrusive visualization of temperature fields, utilizing pyrographite sensing element and IR thermal detection systems seems to be particularly suitable for detection of temperature changes of mobile and immobile media.

In the case of mobile media, above nonintrusive method is able to measure their movement determined by their displacement and/or velocity.

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