

Laser Heating and Surface Optical Monitoring Techniques: A Survey¹

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Laser heating of condensed matter is reviewed according to the temporal duration of the excitation. Differences in the various heating regimes are discussed and relevant diagnostics techniques are briefly illustrated, especially in connection with picosecond and femtosecond laser pulses. The recent results obtained with ultrafast excitation strongly suggest that the extreme condition of excitation of matter may be reached and diagnosed using these techniques.

KEY WORDS: laser heating; optical techniques; surface heating.

1. INTRODUCTION

Heating of solids for testing their material properties at high temperatures is being performed using several techniques. The ideal case of a sample at constant temperature and thermally decoupled from the surrounding media appears to be the best experimental condition one can approach in a laboratory. Unfortunately, this case may occur only for relatively low temperatures. High temperatures, instead, may be reached only for short times on small volumes. In this regime, equilibrium conditions may be approached only asymptotically. Interpretation of the results becomes increasingly complicated by transient effects in connection with the shorter heating times. Laser heating techniques have several unique features in this area of investigations. Energy is deposited on the surface of the sample, and in the usual case of optically dense materials, the depth of the deposition is limited to a fraction of a micrometer. Thus one may speak of surface deposition of the beam energy. Laterally, the beam may be adjusted to the

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size of the sample or restricted to very small areas ($\approx 10^{-4}$ cm²) so that at constant energy the specific power over the surface may be varied by focusing. The other main parameter which characterizes the interaction of solid surfaces with laser pulses is time, which may be varied by nearly 15 orders of magnitude from cw operation to femtosecond pulses. This extreme range makes it possible to observe the same temperature regime on a sample under different excitation conditions and to evaluate the effects connected with the nonequilibrium conditions of the surface being irradiated. When the temporal duration of the laser matches the characteristic times of particular mechanisms, different regimes in the heating and relaxation of the samples are established. On this basis, a classification of the main features of the interaction is possible and is outlined here.

Laser techniques applied to the field of thermophysics have been used extensively in the past, mainly with laser pulses in the millisecond to microsecond range. In these regimes, one may find conditions under which local equilibrium is reached in portions of the sample. Direct information on temperature and material properties may thus be directly inferred via traditional techniques, i.e., pyrometry, electrical probing, etc. With shorter heating pulses, however, the fast kinetics of the interaction requires higher temporal resolution coupled with suitable spatial resolution due to the presence of steep gradients on the sample. Here, optical diagnostics techniques seem best suited to follow and resolve the complex phenomena occurring on the irradiated surface. In this regime, therefore, only indirect information is obtained on thermal properties of the sample via its time-resolved optical response. This procedure unravels the whole kinetics of the heating and relaxation processes in the sample.

In this introductory paper, I briefly illustrate the main heating regimes occurring in the interaction of laser pulses of different energy and duration with solid absorbing samples and some of the diagnostics techniques implemented in connection with laser pulse heating experiments. The accent here is on the less investigated and potentially important domain of ultrashort measurements of thermophysical properties. The future work and possible implications of the latest techniques, relevant for the field of thermophysics, are also addressed.

2. INTERACTION REGIMES

The basic mechanism of energy transfer from laser pulses to matter is via photon-electron interaction. This occurs according to the relation $I(x) = I_0 \exp(-\alpha x)$ describing the attenuation of the laser intensity I at a depth x from the surface of the sample, i.e., within an absorption depth α^{-1} of the radiation in the material. This distance is of the order of $\approx 10^{-5}$ cm

for most metals irradiated by visible radiation. This extremely thin, directly heated layer makes it possible to reach very high energy densities and therefore high temperatures even with modest laser intensities (power per unit surface). Transport mechanisms broaden the heated volume with characteristic time constants. They include electron and heat transport through the sample. Also convection to the buffer gas and irradiation into the surrounding space play an important role in dissipating the heat from the interaction area. At high laser intensities, plasma effects due to ionization of the surface or of buffer gas molecules in front of the irradiated area further complicate the picture. These aspects are well-known and described in detail in investigations of laser produced plasmas. They are, however, beyond the scope of thermophysics and are not illustrated here. The features of the interaction are therefore strongly dependent upon the temporal characteristics of the heating source and are classified accordingly below.

2.1. Continuous Laser Heating

A sample heated by continuous laser radiation will reach an almost-uniform temperature when a balance is established between input laser power and heat loss mechanisms. These include conduction losses to the surroundings through, e.g., support structures, convection losses to the buffer gas around the sample, and radiative losses. At low temperatures, before the occurrence of melting, the main energy dissipation channel for well thermally insulated targets is emission of thermal radiation. This, however, is limited to the blackbody emittance value, which is less than $1 \text{ kW} \cdot \text{cm}^{-2}$ even for temperatures $\approx 3500 \text{ K}$. Therefore, even with small laser powers the melting point of any absorbing material is easily reachable. Above the liquid phase transition, however, the material starts to lose energy also by surface evaporation and steady-state temperature profiles may be obtained throughout the sample. By equating the input power of the laser with the heat per unit time and area removed by evaporation, one obtains in one-dimensional geometry

$$\rho L_v v_s = (1 - R) I_0 \quad (1)$$

where L_v is the heat of evaporation per unit mass, ρv_s represents the mass of material evaporated per unit time, R is the surface reflectivity, and I_0 is the laser intensity. Thus, the evaporation rate increases linearly with laser intensity. The Knudsen equation relates the rate of evaporation to the vapor pressure $p(T_s)$ at the surface temperature T_s :

$$\rho v_s = \frac{p(T_s)}{2\pi k T_s / m} \quad (2)$$

where k is the Boltzmann constant and m represents the molecular weight. An estimate of the vapor pressure may be given through the Clapeyron equation. The result is that surface temperature increases only logarithmically with input laser intensity in this steady-state regime. This confirms the experimental evidence [1, 2] that there exists a range of laser intensities where constant surface temperatures are obtained with evaporation rates compatible with the observation techniques. By increasing the laser input intensity, however, modest surface temperature increases are obtained at the expense of substantial evaporation rates. The problem may be reduced in some instances by the use of high-pressure buffer gases, transparent to the optical radiation. The problem of convection and conduction to the surrounding medium has to be evaluated in this case. Furthermore, hydrodynamic forces driven by evaporation may push the surface material out of the heated area, especially in the liquid phase. At high input intensities the laser radiation may interact directly with the expanding gas and a nonstationary situation sets in. Ionization of the vapor plume in front of the heated surface may occur even at relatively low laser intensities, the result being a strongly absorbing plasma which decouples out radiation from the surface.

2.2. Millisecond to Nanosecond Laser Pulse Heating

With very long heating pulses impinging onto a solid surface, the basic interaction length $\alpha^{-1} = 4\pi k/\lambda$ for a laser pulse at wavelength λ , interacting with a material whose imaginary part of the index of refraction is k , is generally much shorter than the diffusion length $l_{\text{diff}} \approx (Dt_1)^{1/2}$. Here, $D = K/\rho C$ is the thermal diffusivity, with K the material conductivity, ρ the density, and C the specific heat. t_1 is the laser pulse duration. During the laser pulse, heat propagates through l_{diff} and uniform conditions may be assumed in this depths. Very modest laser energies are required to heat considerably the surface of an absorbing solid. The main heat loss at low excitation is represented by heat diffusion into the bulk. A unidimensional picture may well represent the situation when the laser-irradiated area has a size which is considerably larger than the diffusion length. In this case, one may even reach constant temperature conditions during the irradiation when the laser power density is balanced by diffusion. A sample thickness of few millimeters ensures that the back surface is always at the initial temperature during the laser pulse. By assuming that the laser energy per unit surface is absorbed into a thickness of the order of the diffusion length,

one may estimate the temperature rise near the surface by a quantity ΔT as a function of the laser intensity I_0 :

$$\Delta T \approx \frac{(1-R) I_0 \sqrt{t_1}}{\rho C \sqrt{K}} \quad (3)$$

The temperature rise is thus proportional to the laser intensity and to the square root of the pulse duration. This equilibrium regime may be reached with millisecond to microsecond pulses and very high temperatures may be reached with small laser intensities. For example, Eq. (3) predicts that a laser intensity of $0.5 \text{ MW} \cdot \text{cm}^{-2}$ for 1-ms laser pulses would raise the temperature of a tungsten sample to its melting point ($\approx 3400^\circ\text{C}$) or $0.75 \text{ MW} \cdot \text{cm}^{-2}$ would suffice to reach the boiling temperature ($\approx 5500^\circ\text{C}$). By shortening the laser pulse duration to $1 \mu\text{s}$, for example, the latter values become $I_0 \approx 15 \text{ MW} \cdot \text{cm}^{-2}$ and $I_0 \approx 24 \text{ MW} \cdot \text{cm}^{-2}$ in the above two cases, respectively.

When the melting temperature is reached at the surface, a thin liquid layer develops on the surface as soon as the latent heat of fusion is released to the material. Melting occurs heterogeneously by nucleation of the liquid phase [3]. The liquid-solid interface is driven inside the depth of the material by the temperature at the interface, which moves toward the solid when it exceeds the melting value T_m and toward the liquid when the temperature at the interface decreases below T_m . The velocity of the interface is basically determined by the degree of overheating or undercooling of the interface. In the interior of the sample these quantities are determined by heat conduction, so that the kinetics of the interface is determined by the specific power transported by conduction.

Two main considerations may be drawn from this picture in connection with thermophysical applications of these regimes. First, the melting process is per se a nonequilibrium process, thus a situation where a non-zero velocity of the interface exists indicates that surface temperature may evolve in time and thermal gradients inside the sample are present. Surface measurements, therefore, should be performed with appropriate time resolution and are representative of nonequilibrium situations which have to be carefully assessed. Second, impurities or dopants contained in solids could change in due time their relative concentration upon melting, thus affecting the results of thermophysical measurements. Both these effects have increasing importance with shorter laser pulse irradiation. At the short end of this temporal regime overheating of the liquid layer by several tens of degree is easily achievable at the liquid-solid interface even at laser intensities when surface vaporization is negligible. Velocities of the interface

of the order of several meters per second have been measured experimentally with nanosecond pulses [4]. A more precise one-dimensional description may be obtained through the diffusion equation for the temperature profile $T(x)$ along the depth of the sample:

$$\rho C \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left(K \frac{\partial T}{\partial x} \right) + \alpha(1 - R) I_0 \exp \left\{ - \int_0^x \alpha dx' \right\} \quad (4)$$

in which the second term on the left-hand side represents the energy deposition of the laser radiation at depth x . All the material parameters are dependent on local temperature $T(x)$. Here, ρ is the material density, C the specific heat, K the thermal conductivity, α the absorption coefficient, and R the reflectivity at the surface. The boundary condition for Eq. (4) at $x = 0$ may be written as

$$\rho v_s \Delta H_v + \bar{\epsilon} \sigma T^4 + c'(T - T_0) = K \frac{\partial T}{\partial x} \quad (5)$$

where the first term represents the energy rate loss in vaporization, the second the radiant losses, and the third the convection losses. The velocity of the receding surface, v_s , with ΔH_v , the enthalpy of vaporization, determines the vaporization losses, $\bar{\epsilon}$ is the average emissivity, and c' is the coefficient of thermal convection to the surrounding gas.

2.3. Picosecond Excitation

When a laser pulse a few picoseconds in duration impinges on an absorbing surface, the diffusion length l_{diff} during the laser pulse is generally shorter than the absorption length $l_{\text{abs}} = \alpha^{-1}$. The absorbed laser energy is deposited in an extremely shallow layer of material. The corresponding temperature therefore may be rapidly raised to extremely high temperatures, well exceeding the melting or boiling point of the substance.

The picosecond excitation regime is also characterized by the first occurrence of nonequilibrium conditions between the subsystems of the heated material. Finite times for transfer of the optical energy from the electronic system into the lattice [5, 6] start here to be detectable. Indeed, in semiconductors, carrier generation by radiation provides some energy storage in the conduction band also at longer times. In the picosecond regime, the Auger effect, in which carrier recombination results in additional photoexcitation of electrons in the conduction band, provides a temporary energy reservoir. In order to model these effects quantitatively, the usual temperature diffusion equation, Eq. (4), now contains a term related to the energy released by the electronic system into the lattice and is therefore

coupled with rate equations depicting the evolution in time and space of the carrier density [7, 8].

The degree of overheating in the absorbing layer may be such as to drive the melting front to extremely high velocities toward the interior of the target. A limit, however, exists to the propagation velocity of the interface, which may be estimated as the product of the vibrational frequency in the material to the average interatomic distance, i.e., the acoustic propagation velocity ($\approx 10^3 \text{ ms}^{-1}$). During the laser pulse, therefore, the melt front cannot sweep all the heated volume. The heated material beneath the molten region may be at temperatures far exceeding the melting point and still being in the solid state. This interesting nonequilibrium situation occurs in the picosecond time scale before conduction into the bulk sets in and smoothes the temperature gradients. Its direct observation is still a challenge to the ultrafast optical techniques available [9, 10]. The energy of the laser pulse is momentarily stored in the atomic system, which preserves its solid phase.

A final comment to this time regime relates to the evaporation rate from the target at high excitation levels. During picosecond laser pulses there is little time for evaporation to take place, simply because of the finite velocity of the molecules leaving the surface [8]. Thus a temporal window may be found during the laser pulse in which evaporation of the surface is negligible. A precise evaluation of this limit affects critically the interpretation of time resolved optical experiments in the first tens of picoseconds following the peak of the laser pulse. This is especially true when the observed optical response appears as if determined by the presence of an optical opaque medium [11]. The picosecond regime is important for thermophysical applications since, contrary to the interaction with longer laser pulses, it allows unobscured view of the sample even at very high excitation regimes. Several effects, however, complicate the interpretation of the data, especially related to the nonequilibrium conditions of the sample and the extremely fast temporal evolution. Finally, a direct comparison on similar samples of thermophysical data obtained in this and in more traditional regimes remains still to be performed to establish full experimental equivalence of the results.

2.4. Interaction with Femtosecond Pulses

When the laser energy is released to a solid in femtoseconds, the electronic system is the only one capable to respond by switching to highly excited states. In due time thermalization of the electron gas occurs by carrier collisions. Evidence of nonequilibrium electron energy distribution has been obtained in a number of experiments for tens of femtoseconds

following the excitation [12, 13]. The very same concept of electron temperature has little significance during the excitation pulse. After thermalization by collisions, hot electrons are first obtained in a cold, unexcited atomic lattice. Energy is then gradually released to the lattice via electron phonon interaction, typically in a few picoseconds. Thus effects connected with a two-temperature system play an important role in the kinetics of the interaction. In some experiments on semiconductors expansion of the electron gas within the material seems to occur prior to the energy deposition on the lattice [14], so that the initially heated layer is thicker than the absorption length $l_{\text{abs}} = \alpha^{-1}$ [15, 16]. Moreover, in the first picoseconds after the pulse, the lattice cannot expand due to its inertia. The characteristic time for hydrodynamic expansion to take place may be estimated as the ratio of the heated layer thickness to the acoustic velocity, resulting in tens of picoseconds. In this time interval the heated material is essentially confined and extremely high pressures may develop [17]. The extremely high laser intensities of femtosecond pulses may transform the solid material directly into a new state in which most of the electrons of the system are promoted to high energy levels, thus breaking or drastically reducing the bonds between atoms [18]. The intrinsic nature of the material is clearly changed in this phase. The system may evolve either into an ionized plasma or into a hot gas once the release of the hydrodynamic pressure allows thermalization of the system. Further detailed investigations on these points are under way.

This picture demonstrates the intrinsic difficulty in discriminating between temperature and plasma effects in high-intensity femtosecond interaction. Contrary to thermal effects, a plasma may have a very fast onset which may well be in the femtosecond time scale. On the other hand, it may last for long times. Thermal effects are intrinsically slower and therefore may be superimposed on or confused with hydrodynamic effects, such as surface deformation and evaporation.

Femtosecond spectroscopy offers new opportunities to thermophysical investigations since it extends the limits of observations of condensed matter to higher temperatures and pressures. The extension to these regimes of quantitative measurements of thermophysical properties has still to be approached. Nevertheless, ultrafast technology and techniques seem appropriate for this task in the near future.

3. TRANSIENT OPTICAL MEASUREMENT TECHNIQUES

Transient phenomena as the ones described here need some form of temporal resolution in the observation instruments. There are, however,

different experimental approaches to conduct a thermophysical experiment with transient surface heating sources.

The first approach is the natural extension of steady-state experimental techniques and consists in heating the sample as uniformly as possible in order to approach quasi-equilibrium conditions and then measuring the various physical quantities in their evolution when the sample relaxes toward equilibrium with the ambient. The time scale for these measurements has to be greater than the interdiffusion times across the sample. Tailoring of the temporal shape of the laser pulse [19, 2] is performed to increase the time interval in which uniform temperature conditions occur on the sample. This approach requires significant amounts of laser energy available to heat sizable quantities of matter for relatively long (down to microseconds) times in order to assure uniform heating of the whole sample.

A second technique, instead, consists in heating very small areas of surface target with laser pulses well characterized in energy, time, and space. Short laser pulses and relatively small laser energies are required. In this case, therefore, the sample is at all times in a transient regime and measurements are performed with the appropriate time resolution.

Both these approaches require ad hoc diagnostics techniques. When working with long time scales, signals directly generated from the heated sample, such as radiation or particles, may be measured readily with appropriate temporal resolution to deduce directly information on the temperature and other parameters of the material. On the other hand, with short laser pulses the required temporal resolution may be obtained only with optical techniques which allow one to evaluate selected optical parameters. In the remaining part of this section, the main diagnostics techniques used in transient laser heating are briefly discussed.

3.1. Pyrometry

The emission of thermal radiation from heated targets gives the most direct information on its surface temperature. This technique is best employed at high temperatures when the maximum of the emission occurs in the visible or near-ultraviolet part of the spectrum. In these cases transient measurements may be performed down to nanosecond resolution with available optical detector technology. Multiwavelength pyrometers have increased the sensitivity and accuracy of transient measurements in the microsecond scale and are routinely used in laser heating experiments. On the other hand, the new image forming devices may be coupled to pyrometers to provide spatially resolved thermal information on a wide area of the heated samples. Thus, microsecond-resolution thermograms of selected areas of heated samples could become available in the near future.

Given the present limits in temporal resolution for high-gain optical detectors, pyrometry appears to be of use only with nanosecond and longer heating times. The other serious limit of this technique is given by its sensitivity to hot evaporated material emitted by the hot sample. Though corrections to the measured data could, in principle, be made, a reduction of this effect calls for an increase in the buffer gas pressure, when possible, thus reducing the useful range of application of this technique.

3.2. High-Speed Imaging

The advent of ultrafast image processing with framing and streak cameras has extended the temporal resolution of these devices to extremely short times. In the streak mode, temporal resolutions of 1 ps or less are possible commercially available instruments. These techniques are particularly attractive for transient morphological studies, evaluation of damage growth during heating [20], transient dimensional measurements, and surface melting evaluation [21]. Whereas the spatial resolution of these devices does not constitute a limitation, however, the linearity of the intensity, its spatial uniformity, and its dynamical range are still limiting the use to selected applications.

3.3. Reflectometry and Ellipsometry

The measurement of the optical properties of the heated materials provides information on the transient material properties and thus are of interest in thermophysical applications. For example, in many materials the onset of a phase transition to a liquid state is generally accompanied by a discontinuity in, i.e., reflectivity which is readily observable. An exact temporal mark for the phase transition and the threshold laser fluence required for melting is thus deduced from these measurements. A number of physical parameters may be deduced from the reflectivity versus laser fluence curves in the vicinity of the phase transition. Thermal diffusivity and conductivity, melt front velocities, heat capacity, and temperature of the phase transition have been evaluated in a variety of experiments for several materials with nanosecond laser pulse heating experiments. Also, temporal information on the kinetics of the phase transition and its relevant time scales has been obtained. Major impulse to this activity has been the technological relevance of laser annealing of semiconductors and of other materials of relevance for the electronics technology [22].

The optical properties of a material are determined by its complex index of refraction $n = \sqrt{\varepsilon} = n + ik$. Thus, two simultaneous measurements are required to evaluate n . For transparent samples simultaneous transmis-

sion data may be taken. Alternatively, transient ellipsometric measurements may be performed at oblique incidence using, for example, two different detectors for parallel (s) and perpendicular (p) polarizations with respect to the plane of incidence. From s - and p -reflectivities the index of refraction is deduced via the Fresnel formulae and the dielectric constant of the material [23]. Evaluation of these quantities for several wavelengths is important to deduce, through proper modeling, the electronic as well as the lattice contributions and therefore the intrinsic properties of matter at high temperatures and pressures.

In order to improve the temporal resolution of optical measurements beyond the limits of present electronic detectors, one may consider active transient illumination of the heated samples. The principle of stroboscopic imaging finds here an application which pushes the "exposure time" to the ultimate values of a few femtoseconds, i.e., to the pulse duration of ultra-short laser sources [24]. The technique in its simplest form is known as "pump-and-probe" and uses a portion of the heating laser pulse to measure, at a variable and temporally delayed time, the reflectivity of the sample at the center of the heated area. Temporal resolution is thus determined by the pulse duration and timing with respect to the heating laser pulse by the difference in length of the optical paths of the two beams. The result is the reflectivity of the sample during and after the heating process. Improvements to the technique include different wavelengths for the probing beam with respect to the pump pulse. This may be obtained by standard nonlinear optical techniques such as harmonic and parametric generation, Raman shift of the pump pulse in nonlinear media, and, with femtosecond pulses, continuum generation in liquids.

3.4. Nonlinear Optical Measurements

A survey of the optical techniques used in connection with thermophysics must also include nonlinear optical techniques as a diagnostic tool which has been used increasingly in recent years. Second harmonic radiation generated in reflection from heated solids, for instance, has been used [18, 25–28] to monitor the phase transition in noncentrosymmetric crystals such as GaAs during heating. In this case, femtosecond observations have shown the presence of an electronic transformation of the material prior to actual melting due to the promotion of a sensible fraction of electrons to the conduction band of the semiconductor [18]. Stimulated Raman scattering from hot solids has also been used in the past with nanosecond as well as picosecond laser excitation to deduce directly the melting temperature of silicon [29]. It may well be that in the near-future intense femtosecond laser beams could explore the optical nonlinear behavior of materials as a direct means to evaluate physical quantities such as temperature and pressure.

4. CONCLUSIONS

Two main approaches seem relevant in the field of surface heating thermophysics. The first is the use of the laser as a "conventional" heating agent which raises the temperature of the sample to the desired level. Data for relevant thermal analysis are then carried out during stationary conditions of the sample or during the relatively long lasting cooling phase. The second approach, which refers mainly to ultrashort pulse experiments, is to limit the study of the interaction on the central part of the focal area and, from time-resolved optical data, deduce the physical quantities relevant to the thermophysical investigations. This approach, in which equilibrium conditions may not be reached before the onset of hydrodynamical expansion, is motivated by the complexity of the measurement process with a time resolution appropriate to the extremely fast heating and relaxation times, which only recently the advent of ultrafast optical techniques has made possible. The derivation of thermophysical properties requires in some cases ad hoc assumptions in order to overcome the non-local-equilibrium conditions probed at short time delays after irradiation of the sample. On the other hand, other properties (e.g., electrical conductivity) may be directly deduced in some cases by time resolved ellipsometry. Laser heating, furthermore, appears to be a viable solution to explore the extreme range of thermophysical parameters with limited energies locally delivered to small samples. Although measurements in the range below nanoseconds are still of "exploratory nature" [30] and cannot compete in accuracy with measurements performed on longer time scales, nevertheless, they have already clarified several aspects relevant to thermophysics. The need for systematic investigations on short pulse interaction is felt also in comparing experimental results on specific materials and assessing the validity of these methods which rely on transient measurements in the presence of steep spatial gradients.

The richness and complexity of the phenomena occurring in the interaction of laser pulses with solid targets make it a stand-alone topic which extends beyond the limits of thermophysics. However, the versatility of laser experiments and the possibility of changing levels of excitation and time duration over so many orders of magnitude certainly represent a useful tool for deducing material properties at the extreme conditions of interest in modern science and technology.

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