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Issues and Future Directions in Subsecond Thermophysics Research[1]

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The key issues and anticipated future directions in subsecond thermophysics research are presented and discussed. The main emphasis is placed on experimental techniques for measurements of selected thermophysical properties utilizing rapid volume heating (resistive self-heating) and rapid surface heating (laser pulse-heating) methods. The time regime covered is from 1 to 10^{-12} s. Specific research topics and key research areas are identified and discussed.

KEY WORDS: dynamic measurements; high temperatures; laser heating; pulse heating (resistive); subsecond thermophysics.

1. INTRODUCTION

The primary motivations for trying to conduct thermophysical experiments on subsecond time scales are to extend the measurements of thermophysical and related properties to temperatures far above the limits of steady-state methods or to make measurements on systems far removed from thermodynamic equilibrium.

The objective of this paper is to present and discuss the key issues and anticipated future directions in subsecond thermophysics research covering

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the time regime from 1 to 10^{-12} s. The main emphasis is placed on experimental techniques for thermophysical measurements related to rapid volume heating (resistive self-heating) and rapid surface heating (laser pulse-heating) methods. The volume heating methods cover experiments on the time scale from subsecond to submicrosecond, while the surface heating methods cover those from subsecond to picosecond. Specific new research topics are discussed in relation to the presentations of the various techniques. Key research areas are identified in which effort is needed for substantial progress in subsecond measurements and which have unusual promise for useful new scientific results. These areas include equilibration during rapid heating, measurements at higher temperatures combined with higher pressures, measurements on specific materials, measurements of microstructural properties at high temperatures, temperature standards at high temperatures, and limiting instabilities.

2. VOLUME HEATING METHODS

Rapid volume heating methods are developed for the study of electrically conducting materials and involve resistive self-heating of the specimen. These methods are usually classified under two categories depending on the duration of the experiment: subsecond (generally referred to as millisecond resolution) and submillisecond (generally referred to as microsecond resolution). The key issues and future directions in research on the two categories of volume heating methods are presented in the following subsections.

2.1. Millisecond-Resolution Techniques

In the last two decades, millisecond-resolution pulse-heating techniques have provided the most accurate data on selected thermophysical properties of electrically conducting solids from about 1500 K up to the melting temperature of the specimen. Considerable research efforts have gone into the original development of the technique (the simultaneous determination of heat capacity, electrical resistivity, and hemispherical total emittance) and into the subsequent extensions and refinements (normal spectral emittance, thermal expansion, temperature and energy of solid-solid phase transformations, and measurements at the melting point). These measurements have now reached a complete development stage [1–5], with research efforts directed to areas of further potential improvement such as (1) the use of specimens with simpler geometries and with easy-to-check cross-sectional uniformity, (2) better measurements of the radiative heat loss (and hence, of hemispherical total emittance), and the development of pyrometer calibration schemes in which reference is always made to a blackbody temperature scale, even when pyrometric measurements are performed on the surface of the specimen.

The millisecond-resolution techniques still have considerable potential for future growth particularly in areas such as (1) development of new techniques for accurate measurements of additional thermophysical properties and (2) practical *in situ* applications in industrial manufacturing, process control, etc. In research applications, a number of additional properties such as thermal conductivity/thermal diffusivity, velocity of sound/compressibility, and selected mechanical properties could be accurately measured with appropriate modifications to existing pulse heating systems. The challenge in industrial applications will be to develop simplified practical techniques which have a sufficient level of accuracy and to extend the fast temperature measurements to lower temperatures (the region 500–1500 K where most industrial applications are found). The last item requires the development of high-speed pyrometry in the infared (5- to 10-μm region) with challenging new problems of detectors and calibration schemes suitable to that wavelength region.

An important characteristic of millisecond-resolution techniques is that several properties can be simultaneously measured on the same specimen in a single experiment. For example, in the measurement of heat capacity, a correction for heat loss from the specimen due to thermal radiation is made based on hemispherical total emittance determined during the same experiment. The power input data in the same experiment (current and voltage drop) permits the determination of the resistance of the specimen and, when geometrical factors are accounted for, the electrical resistivity. The capability of simultaneously measuring additional properties, as mentioned earlier, will provide a consistent set of data for the determination of other quantities such as the Lorenz number, the dilation contribution to heat capacity, the Gruneisen parameter, etc.

Recent progress in the development of spatial scanning pyrometry may enable rapid measurements of temperature profiles along the length of the specimen. A system which combines a rotating mirror with a high-speed pyrometer has been developed [6] and applied to measurements of thermal expansion [7] and thermal conductivity [8]. Another promising approach, presently under development, involves a high-speed pyrometer in which the detector is a linear photodiode array consisting of multiple elements [9]. When combined with an existing pulse-heating system, this instrument should enable the simultaneous measurement of thermal conductivity, heat capacity, and electrical resistivity on the same specimen.

A future application of millisecond-resolution pulse-heating techniques is the measurement of the velocity of sound. This would provide important data on the elastic constants and the compressibility of solids at high temperatures where reliable data do not exist at present due to limitations of steady-state measurement methods. An attractive feature of such a measurement capability would be that, in a single pulse-heating experiment, one could perform simultaneous measurements of heat capacity at constant pressure, thermal expansion, and velocity of sound (hence, compressibility) from which one could determine the dilation contribution in order to "correct" the measured heat capacity to heat capacity at constant volume, which is needed in any comparison with theory. These measurements would also provide consistent data to determine the Gruneisen parameter.

Other areas for future development are improved methods for the measurement of mechanical properties of metals, such as tensile strength, and the study of tensile instabilities at high temperatures under rapid heating conditions. As yet, relatively little work has been done in this area due, in part, to considerable experimental challenges involved in maintaining the specimen under a constant-load environment and uniform temperature during rapid heating and in measuring the resulting deformations.

Millisecond-resolution techniques also have possible applications in the study of metals in their liquid phase when such measurements are performed in a microgravity environment [10]. When compared with the much faster microsecond-resolution techniques, such an approach has the potential of yielding more accurate thermophysical data under conditions which better approximate thermodynamic equilibrium. The initial challenge in this work is to determine the geometrical stability criteria which will maximize the temperature excursion into the liquid phase when specimens are rapidly melted under microgravity conditions. If successful, this approach should enable the measurement of selected thermophysical properties of liquid metals such as surface tension, heat of fusion, heat capacity, and electrical resistivity. Progress in this direction has been made and preliminary experiments have demonstrated the feasibility of measuring the surface tension of liquid metals at their melting point [11].

2.2. Microsecond-Resolution Techniques

Preliminary efforts in developing microsecond-resolution techniques were begun in the 1970s to meet the challenges of determining the thermophysical properties of liquid electrically conducting materials in the temperature range 2000–10,000 K. Summaries of research conducted in this area in several laboratories are given in the literature [12, 13].

In general, uncertainties in the measurements with these techniques are considerably greater than those with the millisecond-resolution techniques. Unquestionably, more work is required to bring the microsecond-resolution techniques to a mature level.

Although microsecond-resolution pyrometry is reasonably well developed [14], measurement of the specimen's true temperature still presents serious problems due to the difficulty in achieving a blackbody configuration. The machined cavity used for solid specimens in the millisecond-resolution techniques cannot be used for liquid specimens. For specimens large enough to create a cavity, the current requirements of the system would be substantially greater to reach the same states in the given time scale, and the skin effect would require long enough heating times to cause mechanical stability problems. In addition, once the specimen melted, the cavity would not be stable. Its presence would also be a serious perturbation in the geometry, inducing both heating gradients and magnetic instabilities. On the other hand, normal spectral emittances, $\varepsilon(T, \lambda)$, required for calculating temperature from surface brightness are not well determined, particularly for the liquid range. In the absence of $\varepsilon(T, \lambda)$ data, temperature or wavelength independent emittances are sometimes assumed for determining the temperature of molten specimens; however, caution should be exercised in using these assumptions over wide temperature and wavelength ranges.

Another area in need of improvement is the measurement of volume in the liquid range. The usual technique has been the use of streak shadowgraphs of wire specimens, produced by means of back lighting. The requirement of cylindrical shape to convert the shadowgraph measurements to volumes is the key weak point. One possible way to overcome this would be to use thin foil specimens contained by a transparent material so that the geometric shape considered would be better defined. An added advantage is that displacement techniques such as interferometry could be employed. The use of flat slab geometry would also have the potential to overcome a problem with the present technique for measuring sound velocity in the liquid range. A Q-switched laser is used to induce a stress pulse at one side of the liquid column of metal. The arrival of the stress pulse at the opposite side of the specimen is seen in the streak shadowgraph. The measured transit time and distance traveled is limited to the accuracy of the streak shadowgraph. A further problem, however, is that the concurrent stress pulse produced in the containment medium travels around the circumference of the specimen and may arrive at the opposite side before the pulse through the specimen does. Slab geometry would allow delaying this additional wave.

3. SURFACE HEATING METHODS

Rapid surface heating is achieved by radiative energy deposition on the surface of the specimen in short times. In the past, various energy sources, such as a flash lamp or a solar furnace, were utilized. However, since the advent of powerful lasers, virtually all rapid surface heating is accomplished by laser radiation. Laser interaction with matter (both conductors and insulators) has been exploited for heating of surfaces well above their melting point. The short penetration depth of laser light into absorbing materials results in extremely high surface temperatures with low energy requirements. Also, due to the focusing characteristics of laser light, localized heating on specific areas of the specimen is achievable with relative ease. The amount of matter being heated is considerably smaller than with other techniques. Several heating regimes may be used in laser heating experiments depending on material parameters as well as heating levels required [15].

Continuous-wave (CW) laser heating of materials has been extensively used for both physical measurements and surface treatments. Stationary temperature depth profiles may occur when a balance between input laser flux and thermal conduction into the bulk is achieved. At high excitation fluences, however, a balance may be obtained with surface cooling by evaporation of the heated material. In this case, progressive sample ablation occurs with considerable recession velocities. Heating with CW laser beams is now a well-established procedure in various thermophysical laboratories. Measurements of various quantities versus temperature have been performed in a broad range, with or without a buffer pressure gas. Measurements are limited at the highest temperatures by the transparency of the vapor cloud in front of the target, which may prevent optical pyrometry measurements [16].

In the mid to upper millisecond range, pulsed laser heating is used for measuring thermal diffusivity and, in some cases, heat capacity of materials usually in their solid phase and sometimes in their liquid phase near the melting point. The technique for thermal diffusivity measurements is well developed and widely used [17]. However, in this technique, the temperature excursion in the specimen due to the laser pulse is generally small and is above a steady-state ambient temperature; and because of this, it may not be classified as a totally dynamic technique.

In the microsecond range, pulsed laser heating has been used to obtain selected thermodynamic properties of high-melting point materials of interest in nuclear reactor technology at high temperatures. The measurement system is very sophisticated, involving a variety of time resolved electrical, optical, and X-ray techniques [18, 19]. Because of the difficulties in the technique, the measured quantities are subject to considerable uncertainties and their interpretations are not satisfactorily resolved as yet.

In the nanosecond range, the use of laser pulses brings some advantages in terms of efficiency in heating and stability of the heated area after melting. Time-resolved measurements during the heating and rapid cooling of the melt may be performed both via optical means as well as with electric probes. Experimental apparatus involving optical and electrical diagnostics are in operation in many laboratories [20]. In particular, transient reflectivity and ellipsometric experimental methods have been used in several instances to determine the electronic structure of molten materials. Moreover, transient conductivity is being used to monitor the kinetics of the interface during nanosecond laser heating of semiconductors. These techniques are, however, limited to excitations not involving excessive material evaporation, which might affect optical as well as electrical measurements. Surface heating with CW-to-nanosecond pulse laser sources is now a well-established technique and some physical properties of selected elements have been determined. Extension of the investigations to a broad range of solid materials is most desirable.

The ultimate regime in surface heating of solids occurs with picosecond laser pulses, i.e., when laser pulse durations approach the characteristic times for energy relaxation of the photoexcited electron gas to the lattice. The thermophysics field is still at its infancy here, although some experiments have already demonstrated the ample capabilities of this regime [21]. Heating by picosecond laser pulses has several distinctive features. First, heat is being deposited in such a short time that heat conduction out of the light-absorbing volume does not occur within the laser pulse duration. Extremely high temperatures are thus generated in a shallow layer. Second, due to the short excitation times, matter is being heated while still in a quiescent state and may be observed and probed before it escapes out of the irradiated area by evaporation or ablation. Both these circumstances are extremely attractive in the study of high-temperature regimes of condensed matter and open new possibilities in the field of thermophysical measurements. Systematic investigations of thermodynamic properties measured during picosecond transients are not available at present. In the future, comparison between equilibrium and transient experimental data will clarify the limits of validity of ultrafast heating techniques. If consistent results are obtained, then picosecond laser heating may be used with confidence to extend measurements of thermodynamic properties in a temperature range not accessible with other means. In particular, the structure of the liquid phase of high-melting point materials of technological relevance, such as carbon, and their thermodynamic properties may be unraveled. Thus, the need of dedicated ultrafast heating facilities appears to be of increasing priority in the thermophysics community.

4. KEY RESEARCH AREAS

Specific topics and directions in subsecond thermophysics research have already been discussed in the earlier sections in relation to the specific techniques. In this section, key research areas are identified in which effort is needed for substantial progress in subsecond thermophysical measurements.

4.1. Equilibration Problems

In dynamic experiments, two kinds of deviation from equilibrium can occur: systems can be inhomogeneous yet in local thermodynamic equilibrium (LTE), or systems can be homogeneous and out of LTE. Examples of the former are rapidly pulse-heated metals where the skin effect causes inhomogeneous energy deposition, or where radiation cooling of a surface creates a temperature gradient, or where laser energy is absorbed in a very shallow surface layer. An example of the latter is a molecular system in which a vibrational degree of freedom has been excited, by stimulated Raman scattering, for example, to a temperature much higher than the translational degrees of freedom.

The problem of temperature gradients resulting from the skin effect in resistive pulse heating experiments has been dealt with in detail. Careful attention is required in 1- to 10-μs-duration heating to assure reasonably homogeneous samples. The radiative cooling is also a problem but primarily for longer-time scale experiments. If the hot sample is in contact with a good thermal conductor, as in a diamond-anvil cell, for example, with a long heating pulse, or in steady state, the gradients can cause a significant temperature heterogeneity.

Another significant problem associated with systems pulsed to high temperatures is the question of how long a crystal takes to melt and what degree of superheat is possible in a solid. Recent shock-wave measurements indicate that on a nanosecond time scale, solids may be superheated by as much as 500 K [22]. The general problem of the dynamics of melting both in bulk and on surfaces needs quantitative investigation.

A final example of equilibration problems is the dynamics of vapor-bubble nucleation and growth in complex systems, like metals. The long-range interaction in metals complicates the usual nucleation and growth models. For both theoretical and practical reasons, associated, for example, with problems of hypervelocity impact, a quantitative knowledge of vapor-bubble dynamics is required.

4.2. Higher Temperature and Pressure

Combining extreme conditions of temperature and pressure simultaneously presents an experimental challenge. The diamond-anvil cell has allowed some progress and more can be expected. By using special materials, diamond cells can be operated above 20 GPa and 1500 K, and much higher temperatures have been achieved if large thermal gradients are acceptable.

Shock pressures to over 100 GPa are possible with laboratory impact facilities. Associated shock temperatures up to 10,000 K allow us to make thermophysical measurements of reasonable accuracy over a wide range of temperature and pressure. Higher pressures and temperatures require more energetic driving systems, like nuclear explosions, or smaller experiments like those used for laser pulse-driven shock waves. Because of technical difficulties, both of these options will be limited for the foreseeable future.

One recently developed technique to achieve a wider range of temperatures and pressures in a shock-wave experiment involves pulsed preheating and then shocking a sample [23]. These techniques are difficult to control and add inaccuracy to the measurements. The overall usefulness for accurate thermophysical measurements remains to be seen.

One technical area where pulsed heating should be very interesting is in the production of dense nonideal plasmas. These systems, from 0.1 times the critical density to the critical density, have all the complexity of fluids with strong ion correlations, electronic transitions, cluster formation, and plasma phase transitions. Very little is known about the thermophysical details of these dense plasmas.

4.3. Specific Materials and Measurements

Carbon remains as one of the most poorly characterized elements in terms of its basic thermophysical properties. For example, the triple-point temperature of graphite still is uncertain by several hundred degrees kelvin. Another uncertainty is the slope of the diamond–liquid phase boundary, although recent measurements seem to show that it must have a positive dP/dT [24]. The slope of this particular phase boundary tells whether the liquid is more or less dense than the diamond phase, so it is a very basic thermophysical property. Finally, there remains uncertainty about whether fluid carbon is metallic ($\rho \leqslant 100\ \mu\Omega \cdot \mathrm{cm}$), like Si and Ge, or whether there may even be two fluid phases. All of these problems are associated with the extreme temperatures and elevated pressures required to melt carbon, and therefore, the carbon problems represent fundamental challenges to subsecond thermophysical measurements.

With the exception of mercury and the heavier alkali metals, the thermophysical properties of fluid metals, within a factor of two of the critical density ($0.5\,d_c < d < 2d_c$), are poorly measured. We typically have to rely on equation-of-state models calibrated well away from the critical point to establish even this basic thermodynamic point. Critical phenomena in metals may well be qualitatively different from those in simple fluids, since in metals the interatomic potential changes from long to short range around the critical density. Also, the relationship between the critical fluctuations and transport properties is still far from clear. A great deal of work will be necessary to make measurements, necessarily pulsed, at the high pressures and temperatures of most metal critical points. However, the physics of these strongly correlated systems should make the effort worthwhile.

Metals can be heated electrically in a carefully controlled manner. Nonmetals, like minerals and ceramics, present a different set of problems, both technical and scientific. A primary scientific problem for more complex molecular systems is understanding the relationship, if any, between chemical reactions and critical fluctuations at very high temperatures. The technical challenge is to find a way to pulse heat insulators to high temperature. Volume heating by relativistic electron beams has been tried, but complete thermophysical measurements have been difficult and inaccurate, as a result of inhomogeneous energy deposition and electrical noise. Preliminary attempts to use pulsed neutron heating of uranium impurities show another possible approach. By controlling the impurity concentration, one can achieve a reasonably uniform heating. However, pulsed neutron sources of sufficient intensity are rare.

4.4. Microstructural Data

The thermodynamic properties of condensed matter are determined by the interparticle potential and the structure of the atoms or molecules. When systems are pulse heated, through melting, for example, the microstructure obviously changes. More subtle changes associated with the way atoms of one type cluster around particles of the other type in binary systems will also affect thermodynamic properties. Also, the dynamics of formation of equilibrium microstructures may be of interest. These problems may be approached by combining pulse heating with pulsed X-ray or neutron scattering, although very few data of this type are presently available.

4.5. Temperature Standards

In the context of this paper, temperature standards refer mainly to the available techniques at high temperatures for obtaining an accurate temperature scale in the region of interest. Temperature standards can be defined to high accuracy up to the melting points of most of the refractory materials. So long as the material remains solid, a good approximation to a blackbody cavity can be made. The effective emittance of a small hole in the cavity wall is close to unity, and the hole dimensions are stable. Since the speed of light is so high, thermodynamic equilibrium can be established in the cavity, even on submicrosecond time scales. If, however, the cavity material begins to melt, surface tension and gravity will distort the cavity opening, thereby eliminating this approach for temperature determination of materials in their liquid phase.

In the temperature range above 4000 K, where most materials are molten except at extreme pressures, a new way of establishing temperature standards must be developed. Since liquid metals can be held in a stable form in the 4000 to 10,000 K range for several microseconds, a characterization of the temperature and wavelength dependence of the emittance, $\varepsilon(T, \lambda)$, may be possible. Since this function tends to be relatively smooth, it may be possible to fit it for some metals with relatively few, simple polynomials. A large number of radiance data points and a detailed sensitivity analysis would be required to determine the potential accuracy of this approach.

A particularly interesting aspect of determining $\varepsilon(T, \lambda)$ is that any deviations from a relatively smooth functional dependence will indicate structure in the joint density of electronic states. Although the loss of long-range order in the fluid metals eliminates many of the critical points in the electronic density of states, there remains some structure which may be observed in reflectance or, equivalently, in emittance. The use of this "emittance spectroscopy" may provide useful new data on the electronic structure of fluid metals for which reflectance measurements are difficult or impossible.

4.6. Limiting Instabilities

For the microsecond-resolution measurements, the upper limit on the useful data has been determined by the onset of complex geometry due to instabilities. It seems likely that the limiting instability is different for different metals. One possibility is superheating into the liquid–vapor coexistence region. Isotherms in the P, V plane, calculated using the classical Van der Waals equation of state, show loops in the two-phase region. The well-known Maxwell construction can be used to determine the fraction of each phase present at each volume. The locus of points where the isotherms have zero slope define the spinodal. For the region between the two branches of the spinodal, the isotherms have positive slope. The compressibilities thus have reversed sign. Increases in pressure resulting from further heating will produce rapid expansion. The boundaries of the two-phase region where boiling or condensation begins are called the binodal. For the case of lead, superheating to the spinodal seems a likely explanation for the instability, but for aluminum, unstable behavior occurs even before the binodal is reached [13], so another mechanism must be involved. A theoretical treatment has been advanced [25] which predicts a plasma phase transition to a nonmetallic state in aluminum at high temperatures, for densities below the critical density. This is the region where unstable behavior is observed in the isobaric expansion measurements [13]. Subsequent work [26] has been done using shock waves to heat porous aluminum to states where the release path in phase space will pass through the region where the plasma phase is predicted. The experimental results, however, do not support the existence of the plasma phase. In fact, only indirect experimental evidence exists as a basis for believing in plasma transitions [27, 28].

5. SUMMARY AND CONCLUDING REMARKS

The issues and future directions in subsecond (1–10^{-12} s) thermophysics research have been discussed in the context of measurements of thermophysical properties of materials, mainly at high temperatures. Emphasis has been placed on experimental techniques related to rapid volume heating (resistive self-heating) and rapid surface heating (laser pulse heating) methods. The thermophysical properties that may be measured by the dynamic (subsecond) heating methods are listed in Table I.

For the experiments in the 1-ms to 1-s range, techniques are generally well developed to perform accurate (1–3%) measurements of selected thermophysical properties up to the melting temperature of the specimen. The measured properties have been invaluable in many applications related to high-temperature technologies, including aerospace, nuclear energy, and the establishment of standards (materials and temperature reference points). However, these techniques are too slow for experiments that exceed about 5000 K or where the specimens melt.

For the range 1 μs to 1 ms, techniques are sufficiently developed for measurements on both solid and liquid specimens to yield selected properties data with reasonable uncertainty (3–10%), under favorable conditions. The limited number of measurements have provided useful information on the general behavior of materials, especially in the liquid phase at high temperatures. However, significant improvements in the systems and in the

Table I. Thermophysical Properties that May Be Measured by Dynamic (Subsecond) Heating Methods

Heating method	Time resol.	Solids	Liquids
Volume heating (resistive)	ms	Heat capacity Thermal expansion Electrical resistivity Hemisph. total emittance Normal spectral emittance Solid–solid trans. temp. Solid–solid trans. energy Melting temp. (T_m) Radiance temp. at T_m Thermal conductivity[a] Mechanical properties[a] Velocity of sound[a]	Surface tension Heat of fusion[a] Heat capacity[a] Electrical resistivity[a] Hemisph. total emittance[a]
	μs	(Properties listed under liquids can also be measured for solids near the melting point)	Heat of fusion Heat capacity Electrical resistivity Thermal expansion Velocity of sound
Surface heating (laser)	μs	(Properties listed under liquids can also be measured for solids near the melting point)	Heat of fusion Heat capacity Density Normal spectral emittance Melting temperature Vapor pressure
	ns	Reflectivity	Reflectivity
	ps	Reflectivity Velocity of sound	Reflectivity

[a] At developmental stage.

measurements, especially in temperature determinations, are essential to bring these techniques to a mature level.

For the range 1 ps to 1 μs, the techniques are of an exploratory nature. The measurements are not well understood, the systems are typically out of equilibrium, and many of the measurements are postmortem and, therefore, inferential. But it is just this range in which small total energies can give high-power fluxes to modify seriously very small specimens. The interest by industry in using pulse heating on this time scale, for surface modification of materials, for example, makes developments in this area particularly important.

The dynamic techniques, although in their infancy, have already demonstrated their unique capabilities in relation to measurements of thermophysical and related properties of materials under extreme experimental conditions which often are impossible to achieve by other techniques. Future research in subsecond thermophysics is likely to concentrate on continued development of new and accurate dynamic measurement techniques in all the time regimes, especially in the microsecond and shorter domains. The use of these techniques in property measurements and accompanying theoretical work will significantly contribute to our understanding of the structure and behavior of matter under extreme conditions of temperature, pressure, etc., and under conditions far removed from thermodynamic equilibrium.

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