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Ellipsometry in the Study of Dynamic Material Properties[1]

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Measurements of the time-dependent absolute temperature of surfaces shocked using high explosives (HE) provide valuable constraints on the equations-of-state (EOS) of materials and on the state of ejecta from those surfaces. In support of these dynamic surface temperature measurements, techniques for measuring the dynamic surface emissivity of shocked metals in the near infrared (IR) are being developed. These consist of time-dependent laser ellipsometric measurements, using several approaches. A discussion of these ellipsometric techniques is included here. Ellipsometry permits an accurate determination of the dynamic emissivity at a given wavelength, and may also provide a signature of melt in shocked metals.

KEY WORDS: ellipsometry; emissivity; infrared; pyrometry.

1. INTRODUCTION

HE-shocked surface temperatures lie in the range of 0.04 to 0.2 eV, corresponding to shock heating of surfaces to temperatures from about 400 to 2000 K. This is equivalent to IR wavelengths from about 1.5 to 7 microns. The dynamic (and, in some cases, static) emissivities over these wavelength ranges are not well known, and can only be approximately inferred from multi-channel pyrometer measurements, limiting the accuracy of these

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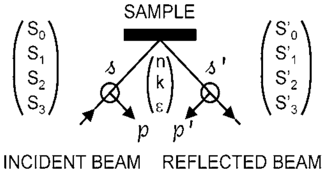

Fig. 1. Here a laser beam in a known polarization state **S** from a polarizer is reflected off a surface. s and p are the linearly-polarized light components perpendicular and parallel, respectively, to the plane of incidence. The polarization state **S**′ of the scattered or reflected beam is measured in the analyzer. Knowledge of both **S** and **S**′ permits an inference of the optical properties (n, k) of the sample surface at the laser wavelength.

temperature measurements. Ellipsometry, or equivalently polarimetry, [1, 2] permits a complementary determination of the dynamic emissivity by measuring the real and imaginary parts of the index of refraction. A graphic depiction of laser ellipsometry is shown in Fig. 1. These emissivity measurements can be used with pyrometer data at similar wavelengths to calculate true temperatures. Dynamic emissivity data may also provide an indication of phase changes. Once the emissivity is derived at one wavelength, a multi-channel pyrometer permits determination of the emissivity at all the other pyrometer wavelengths, since the true or thermodynamic (as opposed to radiance) temperature is independent of wavelength.

2. EMISSIVITY MEASUREMENTS

Typical spectral behaviour of emissivity with wavelength at room temperature is shown in Fig. 2 [3]. Some multichannel pyrometric temperature studies of dynamically shocked surfaces utilize emissivities bounded at the lower end by the static value and at the higher end by 1, the largest possible value. Depending on the material and the speed of the shock, this may or may not be valid at a phase transition. For example, in the case of a non-magnetic niobium wire, explosively driven by high current, the emissivity drops through melt. This can be seen in Fig. 3 [4]. In the case of a high-current driven magnetic nickel wire, on the other hand, at melt the emissivity is seen to rise, as shown in Fig. 4 [5]. Here the Kerr effect, or the introduction of birefringence proportional to the square of the shock-induced electric field, has been ignored. However other factors besides the Kerr effect can drive the emissivity up during shock heating. For example, surface roughness will certainly increase the emissivity, in the absense of melt. With increasing surface roughness the validity of ellipsometry needs to be corroborated using the integrating sphere technique. In addition, dynamic emissivity may be a more sensitive indicator of melt than the radiance temperature.

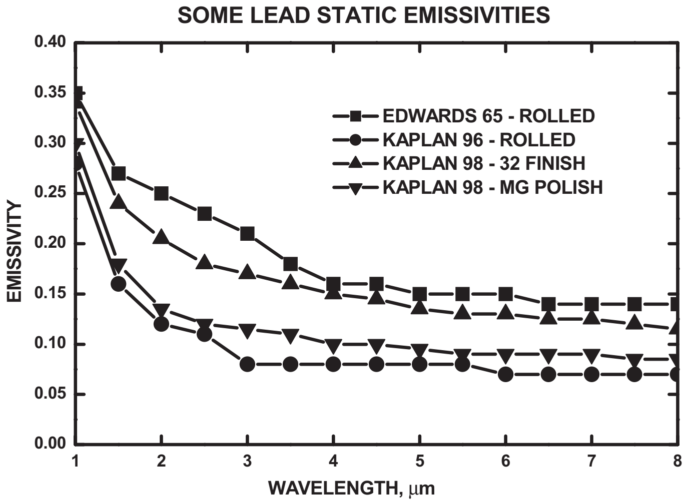

Fig. 2. Note the large variation of absolute values, depending on the surface finish. Compared here are two rolled finishes with a 32-microinch finish and a metallographic polish [3]. Note also here that $d\varepsilon/d\lambda < 0$ while $d^2\varepsilon/d\lambda^2 > 0$.

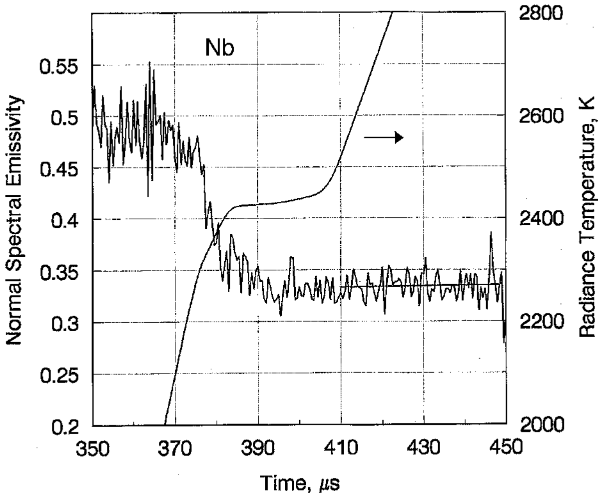

Fig. 3. Decrease of normal spectral emissivity at 677 nm at the melt transition in an exploding non-magnetic niobium wire. The radiance temperature at 656 nm assumes unity emissivity.

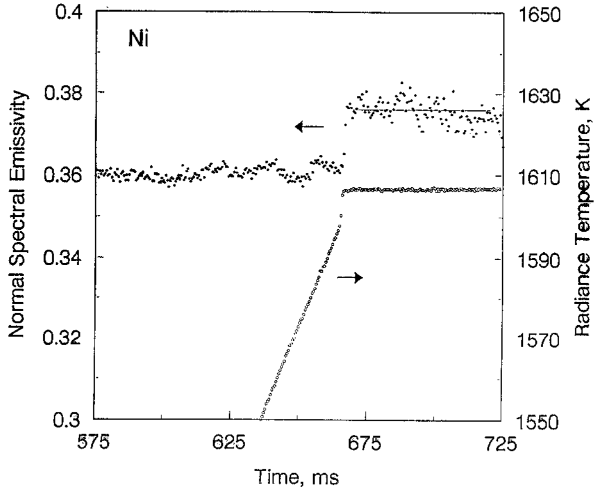

Fig. 4. Increase in normal spectral emissivity at 633 nm at the melt transition in an exploding magnetic nickel wire.

3. EXPERIMENTAL TECHNIQUES

Three ellipsometer instruments are being developed using three somewhat different approaches. All three are currently operating at 1550 nm, which is about the longest wavelength at which both visible-optics work and industry-standard lasers are readily available. This relatively short wavelength should still permit some overlap with pyrometric temperature measurements below 1000 K. Plans are underway for wavelength doubling which will permit ellipsometric measurements at 3100 nm wavelength, a region more centrally located for the 500 to 1000 K temperature regime of HE shocks. Amplitude modulation at the time of interest helps distinguish signal from background light. In the presence of shock background light, an ultimate modulation frequency goal is 1 GHz with 12-bit recording for 1000:1 effective dynamic range. However, it will be some time before this speed at that dynamic range can be realized.

A commercial reference instrument has been purchased from Containerless Research (CRI) in Evanston, Illinois. This instrument has been upgraded to meet our demanding field requirements and is being tested. A conservatively built four-channel instrument, some of its features are as follows. Laser power is currently 2 W. Modulation frequency is at present 50 MHz, and is limited by the following considerations. The four-channel sampling frequency, always twice the modulation frequency, is at present limited to 100 MHz at 12 bits. Also, large-area 2-mm diameter InGaAs

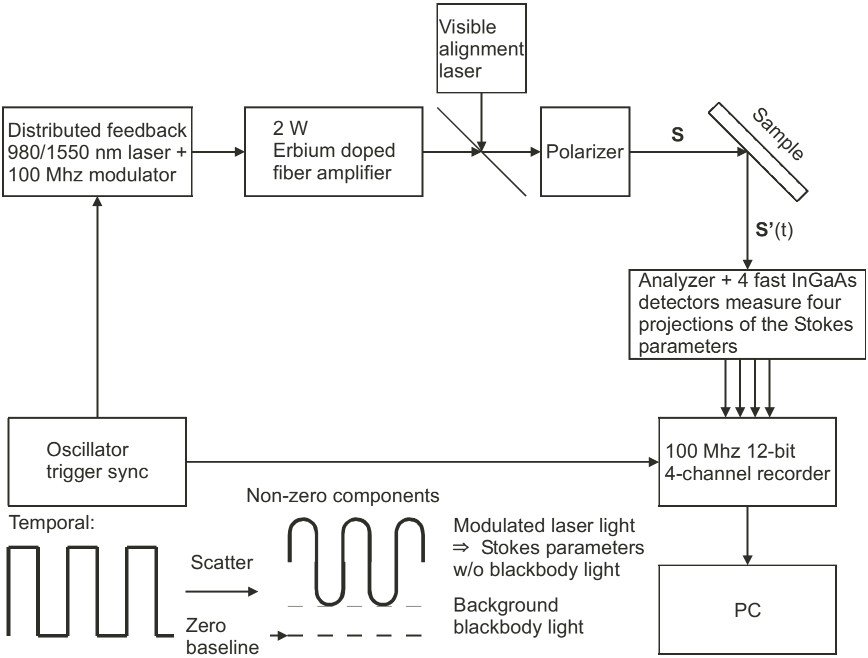

Fig. 5. Schematic of CRI four-channel ellipsometer.

detectors in these dynamic experiments are limited to 100 MHz. Underfilling smaller, and faster, detectors may not accommodate shocked-sample motion. This instrument is shown schematically in Fig. 5.

A second field instrument is being assembled with slightly different features. Instead of four channels it will have six balanced channels, offering some redundancy in remote field operations under adverse conditions. Using smaller fiber-coupled PIN-diode detectors, a modulation frequency of 200 MHz has already been tested with stacked 8-bit recording. The laser power output is 1.2 W at 1550 nm. Figure 6 illustrates this ellipsometer schematically.

A third instrument is being assembled at Bechtel Nevada's Special Technology Laboratory (BN/STL) in Santa Barbara, California. This instrument also uses three channel-pairs, for a total of six channels. The BN/STL instrument will have several noteworthy features. Polarization effects in beamsplitters will be avoided by using near-normal incidence. As in the second instrument, complementary outputs provide background-light and common-mode noise suppression. Minimizing the number of surfaces avoids unwanted reflections. Equal coupling is only required

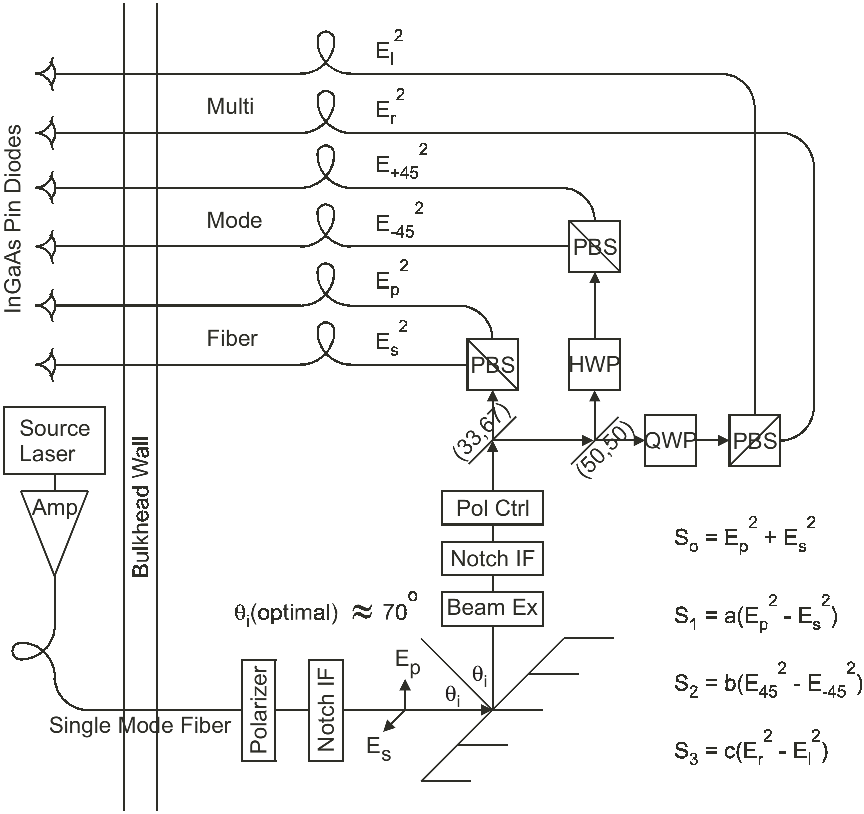

Fig. 6. Schematic of LANL six-channel ellipsometer.

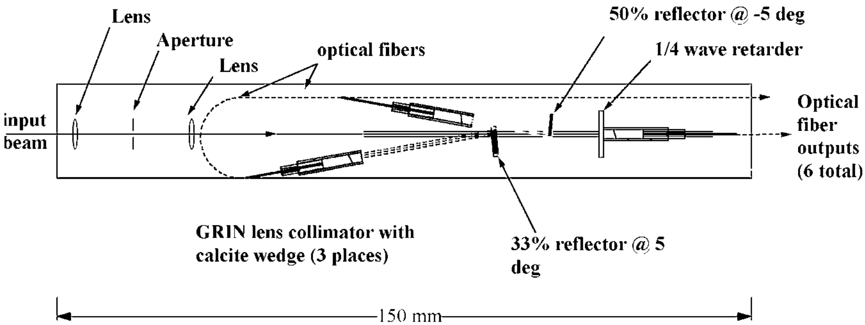

Fig. 7. Schematic of BN/STL six-channel ellipsometer.

within each pair of fibers, not between all pairs. Finally, the construction will be simple and stable to avoid adjustments in the field. Laser power at 1550 nm will be 2 W. Here also stacked 8-bit recording will be used. The BN/STL instrument is shown schematically in Fig. 7.

4. MEASUREMENT UNCERTAINTIES

Sources of uncertainty include alignment errors and polarization impurities as well as noise sources. Also, there may be incomplete or incorrect polarization of the probe beam, probe beam laser noise, calibration errors, digitizer clocking errors and jitter, electronics noise, and possible depolarization of the probe beam by the sample roughness so that the analytical form of the Mueller matrix is not applicable. Furthermore, noisy

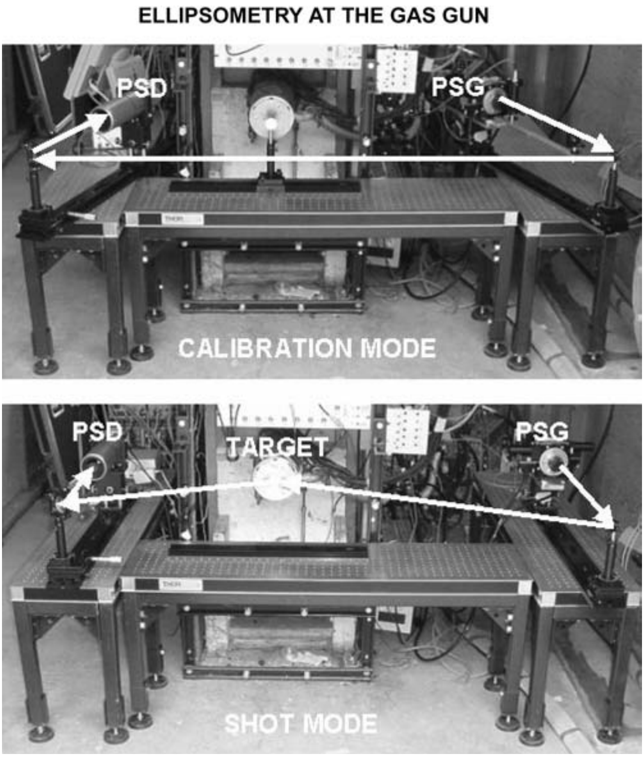

Fig. 8. CRI reference ellipsometer at the single-stage gas gun. Here PSG and PSD refer to the polarization state generator and detector, respectively. After calibration and before the shot, the two rails are toed back out. Nothing is then touched above the rails.

signals may result from the geometry of the measurement, as in the case of exploding wires or loss of surface reflectivity at shock breakout in the case of explosively shocked surfaces. Nevertheless, the ongoing program of modeling the CRI ellipsometer, for example, indicates that typical errors, noise, and imprecision produce tolerable errors in the output.

5. GOALS AND SUMMARY

Near- and long-term objectives include acquisition of emissivity data at near-IR wavelengths for materials of interest. These should improve the accuracy of pyrometric temperature measurements. Variation of emissivity may provide a signature of phase transitions, spall, and ejecta characteristics. The use of LiF anvils is being explored. Also, the validity of the ellipsometric technique as a function of surface roughness is being checked against reference integrating spheres. Comparison of diamond-anvil measurements at static high pressure and temperature with shock conditions at the same high pressure and temperature will be interesting, since the crystal structure may be different in the two cases.

A wide variety of diagnostics is anticipated for these instruments. Local testing will include co-operative efforts in neutron resonance spectroscopy (NRS), laser shock-physics, gas-gun shock experiments, HE-shock tests at BN/STL, and exploding-wire work. Shown in Fig. 8 is the CRI instrument setup at the local single-stage gas gun.

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