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Transverse depth-profilometric hardness photothermal phase imaging of heat treated steels

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A method to image near-surface hardness profiles of heat-treated case-hardened steels using laser infrared photothermal radiometric phase imaging is described. It is shown that thermophysical and mechanical transverse inhomogeneity profiles in industrial case hardened steel samples are well correlated. Phase surface scanning imaging leads to a practical criterion for assessing transverse hardness homogeneity. A simple method based on phase imaging is proposed as a quantitative criterion to determine which steel samples should be rejected for thermal-wave depth-profilometric reconstruction of thermal diffusivity or conductivity.
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INTRODUCTION

Industrial steels are often treated by the addition of carbon and nitrogen, followed by a quenching process in order to produce parts with a tough case. During the heat-treating process, it is possible that a particular sample develops undesired lateral (transverse) hardness inhomogeneities, in addition to the desired depth inhomogeneity.

Much work has been done on the non-destructive evaluation of metals using thermal waves. The field of nondestructive evaluation with thermal waves has been reviewed by Busse.[1] In another review, Busse and Walther reviewed the photothermal nondestructive evaluation of various materials.[2] In particular, thermal waves have been used to determine the depth of structures in metals, for the detection of faults and also for determining thicknesses and properties of hardened layers. Jaarinen *et al.*[3] determined the variation of thermal diffusivity with depth. For metals, the evaluations appear to be limited to detection of areas of prior deformation, fault inspection, profile analysis of seams, and depth profiling. It was also shown that photoacoustic phase angle scanning can be used to measure subsurface structure in metals.[4] The scanning methodology used permitted the identification of small holes hidden under a carefully ground surface of an aluminum sample. While the use of thermal waves to investigate various properties of metals is not new, the present imaging method to investigate the degree of transverse hardness homogeneity in a metal and to use it as a criterion to define thermomechanical homogeneity does not appear to have been previously considered.

EXPERIMENTAL AND RESULTS

The experimental setup for photothermal radiometric (PTR) hardness imaging is shown in Fig. 1. It consists of a high-power semiconductor laser the output of which is current-modulated $I(\omega)$. The beam is expanded, collimated and then focused onto the surface of the sample with a spot size of 1 mm. The harmonically modulated infrared (Planck) radiation from the optically excited sample surface is collected and collimated by two off-axis paraboloidal mirrors and then focused onto a HgCdTe detector. The signal from the detector is amplified by a low-noise preamplifier and then sent to a lock-in amplifier which is interfaced with a PC. Cylindrical samples 1 in. in diameter and 1 cm thick were cut from a bar of AISI 1018 steel. The chemical composition of this steel is 0.15%–0.2% C and 0.6%–0.9% Mn. The surfaces of the samples were ground with a 44, 54, 60 grit sand-wheel. The samples were subsequently carbonitrided and thermally quenched. The samples were treated in a Surface Combustion Super 30 allcase furnace, equipped with a top cool chamber for slow cooling processes. For the carbonitriding process, a base atmosphere consisting of nitrogen and methanol was used. The aim was to produce a nominal carrier gas composition consisting of 40% nitrogen, 40% hydrogen, and 20% CO. To this base atmosphere an addition of enriching methane (CH_4) and ammonia (NH_3) gas was used. The ammonia was present throughout the entire cycle.

A photograph of a carbonitrided 1018 steel sample is shown in Fig. 2. It is clear from the photograph that the surface of this particular sample contains some nonuniformities in color. These optical nonuniformities were generated at locations which were in contact with the basket supporting the steel samples during the furnace processing cycle. Therefore, the question arises as to whether this inhomogeneity is purely optical (confined to the surface) or if it represents hardness inhomogeneities at greater depths. To this end, the surface of the sample was scanned at 10 Hz using infrared photothermal radiometry. The results were mapped and are shown in Fig. 3. Since the amplitude of the signal is proportional to $(1-R)$, where R is the surface reflectance,[5] variations in the signal amplitude over the sample surface resemble the shape of the discolored region of Fig. 2. However, alongside the variation in signal amplitude over the sample surface there is a variation in signal phase as well. The photothermal phase is independent of the factor (1

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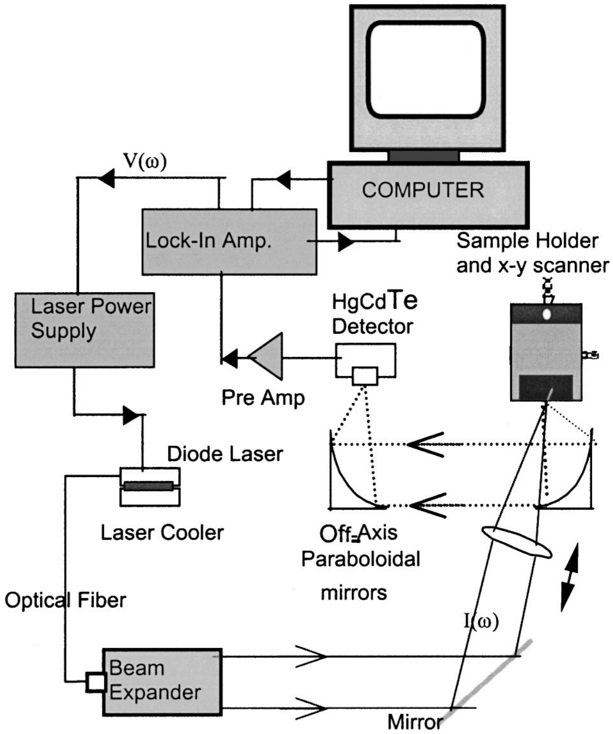

FIG. 1. Instrumental setup.

$-R)^2$ and thus is independent of the optical properties of the surface. The contrast in the phase scan of Fig. 3 suggests the possibility of thermophysical and/or mechanical inhomogeneities across the sample.

Over a range of phase angles, the exact relationship between surface value of the thermal wave field and thermal diffusivity, α, for a homogeneous semi-infinite solid is given by

$$T(\alpha;\omega_0)=\int_0^\infty \frac{1}{\sqrt{\lambda^2+\sigma^2}}\exp-\frac{\lambda^2 d^2}{4}\lambda\, d\lambda, \quad (1)$$

where $\sigma^2=i\omega_0/\alpha$, ω_0 is a fixed angular frequency, and d is the diameter of the beam.[6] Since T is a complex number, we only consider the phase dependence on α. However, for a

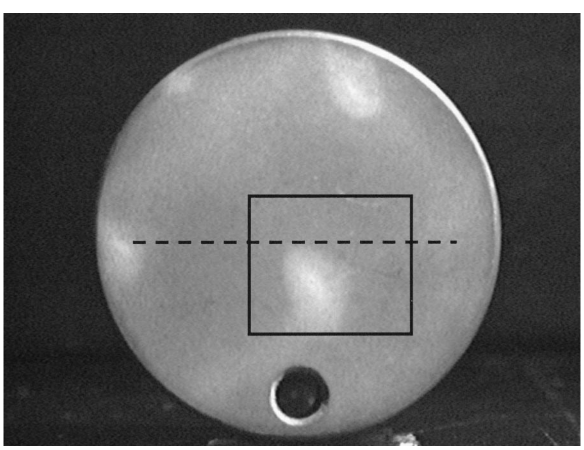

FIG. 2. Example of a nonhomogeneous AISI 1018 steel sample and boundaries of PTR mapping range. The dashed line indicates the locations of PTR and mechanical hardness scans as shown in Figs. 8 and 9.

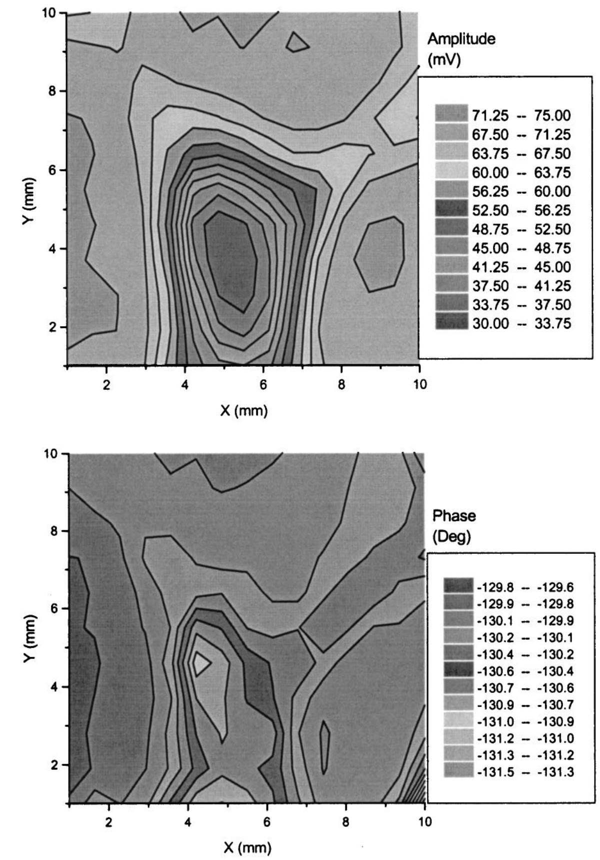

FIG. 3. PTR surface maps of framed area in Fig. 2 with 10 Hz modulated beam (nonhomogeneous sample).

small range of phases such as those found in the phase maps, the relationship between the phase of T and thermal diffusivity, α, can be accurately represented as being linear; $\arg\{T(\alpha;\omega_0)\}=c_1\alpha+c_2$, where c_1 and c_2 can be found by a simple linear fitting. This is accomplished by plotting the phase of Eq. (1), given a particular frequency and beam size, against a small range of thermal diffusivity centered around a chosen reference thermal diffusivity value. In this work, the measured value of thermal diffusivity of the bulk was used. The linearity can be exploited to quickly determine the change in *average* thermal diffusivity across the surface of a sample. At the chosen points, this gives a measure of the mean thermal diffusivity within a thermal diffusion length. A surface map of average thermal diffusivity of the sample in Fig. 3 is shown in Fig. 4. A change in the chosen reference thermal diffusivity value will change the absolute thermal diffusivity values as given in Fig. 4 but will preserve the relative changes in thermal diffusivity.

For comparison purposes, a similar phase/amplitude mapping was performed on a visually homogeneous sample and is presented in Fig. 5. From the very small signal (amplitude and phase) variations across this map, the homogeneity of the sample is obvious. Figures 6 and 7 show additional surface scans performed on yet another sample. This sample is made of AISI 8620 steel, carburized and quenched in a heat treatment similar to that for the 1018 steel. The chemical composition of AISI 8620 steel contains 0.18%–0.23% C, 0.70%–0.90% Mn, 0.40–0.70 Ni, 0.40%–0.60%

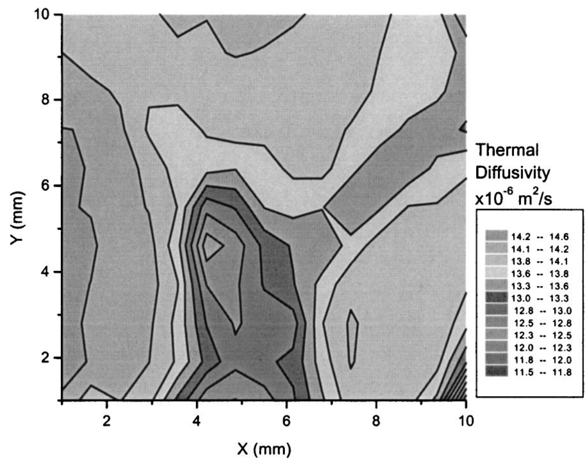

FIG. 4. Average thermal diffusivity surface map of framed area in Fig. 2.

Cr, and 0.15%–0.25% Mo. This sample is inhomogeneous and this can be seen quite well in the phase mappings performed at both 10 Hz and 1 kHz. It should be noted that low-frequency phase variations of up to 2° constitute severe near-surface thermophysical inhomogeneity and possibly strong hardness inhomogeneity, Figs. 3, 6, and 7, whereas phase variations of less than 0.5° are benchmarks of a thermophysically and possibly mechanically homogeneous sample, Fig. 5. Figures 6 and 7 show that amplitude scans are dominated by changes in optical reflectance and thus images obtained at different frequencies vary little. On the other hand, phase images can vary significantly as they are dominated by thermophysical properties within depths on the order of the thermal diffusion length. Although optical inhomogeneities (amplitude images) can be indicators of thermomechanical inhomogeneities (phase images) as in Fig. 3, this is not always the case (e.g., Figs. 6 and 7).

To illustrate the foregoing observations and correlate mechanical and thermal-wave image profiles, PTR radial line scans were performed on a fixed radius of the sample of Fig. 2 at three different frequencies: 10, 100, and 1000 Hz. At each frequency, a 1 mm size beam was employed and measurements were made at eight different points across the radius indicated by dashed lines in Fig. 2. The PTR amplitude and phase signal results are shown in Fig. 8. The shape of the signal amplitude across the scanned diameter remains essentially the same for the three frequencies, an indication that the surface optical reflectance dominates the PTR amplitude. However, the shape of the signal phase changes with frequency and thus points to changing thermal properties of the sample across the transverse scan. The implication here is that PTR phase imaging such as shown in Figs. 3(b), 6(b), and 7(b) is capable of yielding subsurface depth profilometric hardness images at different frequencies.

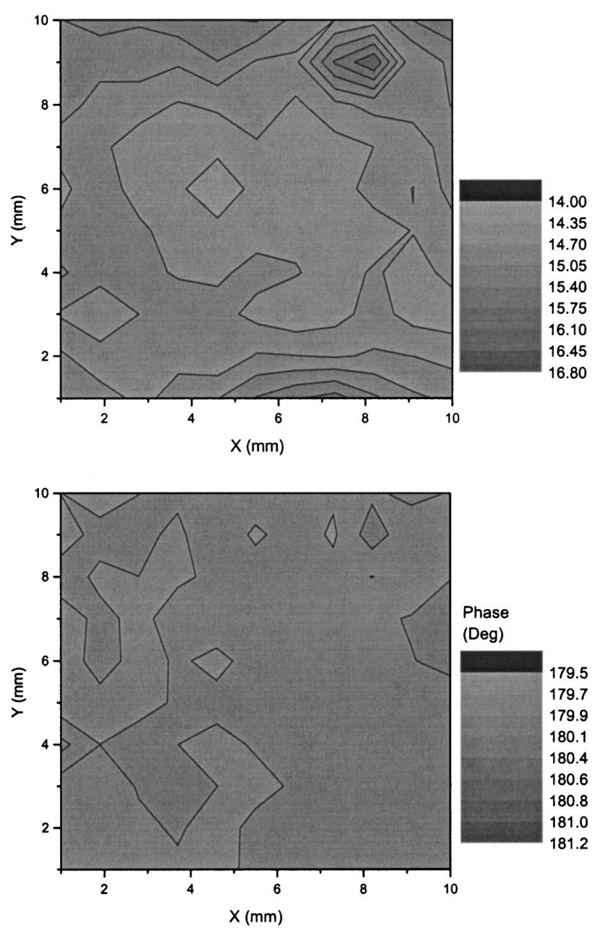

FIG. 5. PTR surface maps of a homogeneous AISI 1018 steel sample with 10 Hz modulated beam.

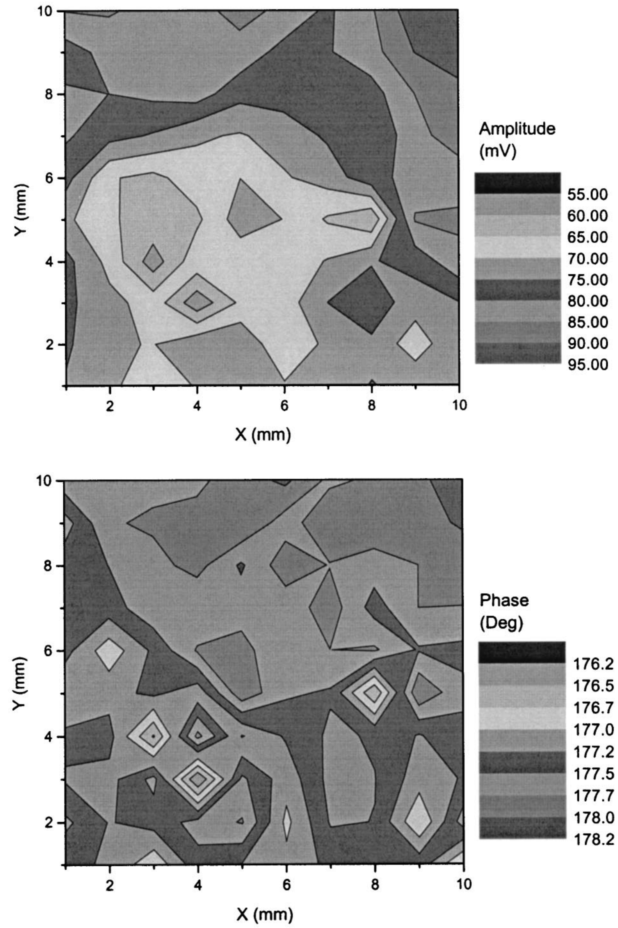

FIG. 6. PTR surface map of a nonhomogeneous AISI 8620 steel sample with 10 Hz modulated beam.

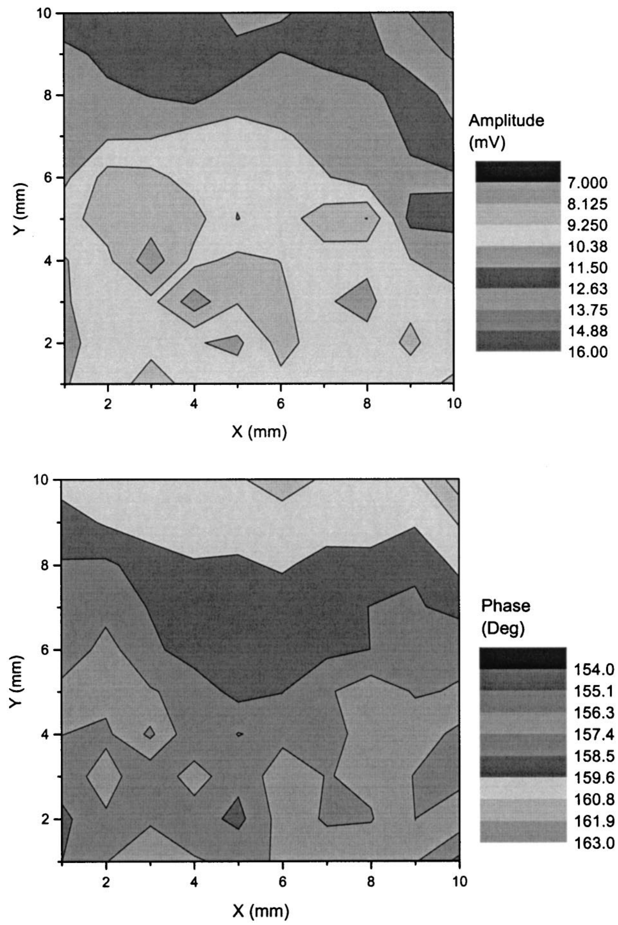

FIG. 7. PTR surface map of a nonhomogeneous AISI 8620 steel sample with 1 kHz modulated beam.

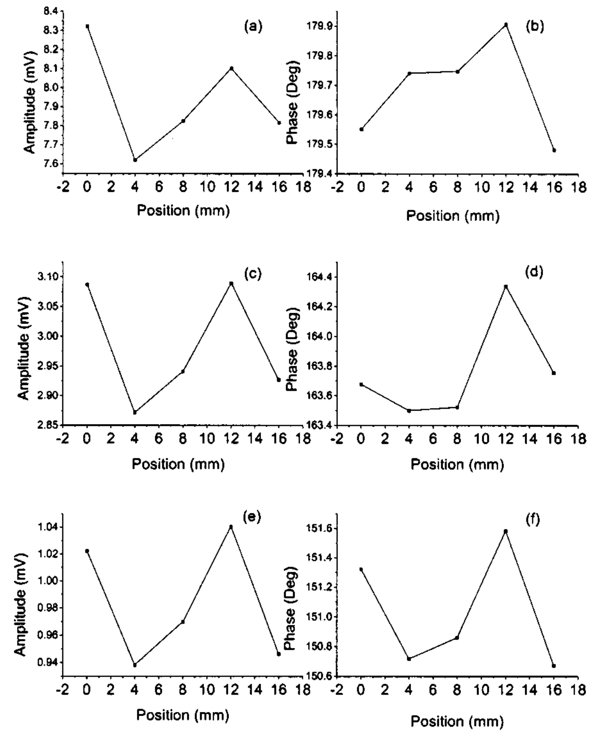

FIG. 8. PTR line scan across a particular sample of AISI 1018 steel: Modulation frequency: (a) and (b) --10 Hz, (c) and (d) --100 Hz, (e) and (f)—1000 Hz.

To demonstrate this claim, the corresponding surface microhardness tests of the same sample were made along the dashed line of Fig. 2 with an indenter and are presented in Fig. 9, confirming that the chosen sample does indeed contain a transverse inhomogeneity. In particular, there is a clear correlation between the diameter hardness test performed with a 1 kg load [Fig. 9(a)] (which thus indents deeper into the steel) and the phase portion of the line scan performed at 10 Hz. This corresponds to a thermal diffusion length of roughly 700 μm, assuming a thermal diffusivity value for the AISI 1018 steel of 1×10^{-5} m^2/s (Ref. 2, Table I, p. 219). There is also a good correlation between the transverse hardness test performed with a 300 g load (shallower indentation) and the phase of the line scan at 1000 Hz which probes within a thermal diffusion length of approximately 70 μm. For further evidence of transverse inhomogeneity, a sample was chosen for destructive microhardness depth measurements to be performed at various locations along the diameter, as indicated in the inset of Fig. 10. The results of this test are shown in Fig. 10. From this figure, it is clear that the microhardness at a particular depth is not uniform across the sample, varying as much as 120 HV. Therefore, the sample contains a transverse hardness inhomogeneity, the profile of which is well correlated with the thermophysical inhomogeneity profile imaged by the PTR phase, Fig. 8. In view of the established correlation between thermophysical scan profiles, represented by the thermal-diffusion-length/depth-averaged thermal diffusivity and the hardness scan profiles, Figs. 8 and 9, it can be concluded that the PTR phase images of Figs. 3–7 can be used as hardness images. The changes observed between the low-frequency phase image, Fig. 6, and the high-frequency image, Fig. 7, can therefore be interpreted as variations in hardness profiles, roughly averaged over one thermal diffusion length.

DISCUSSION

The present experimental results show that thermal-wave phase hardness scanning imaging at a fixed modulation frequency constitutes a powerful criterion for transverse homogeneity assessment within one thermal diffusion length in hardened steels. Among many potential uses of such a criterion, from the photothermal point of view it can be applied in selecting suitable candidates for thermal diffusivity and/or conductivity depth profile reconstructions in case hardened steels.[7] Depth profilometry is a thermal-wave inverse problem where thermal diffusivity depth profiles are reconstructed from surface experimental data as a function of modulation frequency. Theoretically, the problem requires one dimensionality and thus a large incident beam spot size is used so that lateral diffusion can be neglected. Numerical algorithms exist that allow for the reconstruction of the thermal diffusivity depth profiles from surface frequency dependent measurements.[8,9] Since most thermal-wave inverse problem numerical algorithms developed to date are applicable to the one-dimensional problem only, a one-dimensional geometry is required experimentally, including samples with material inhomogeneity in the depth direction

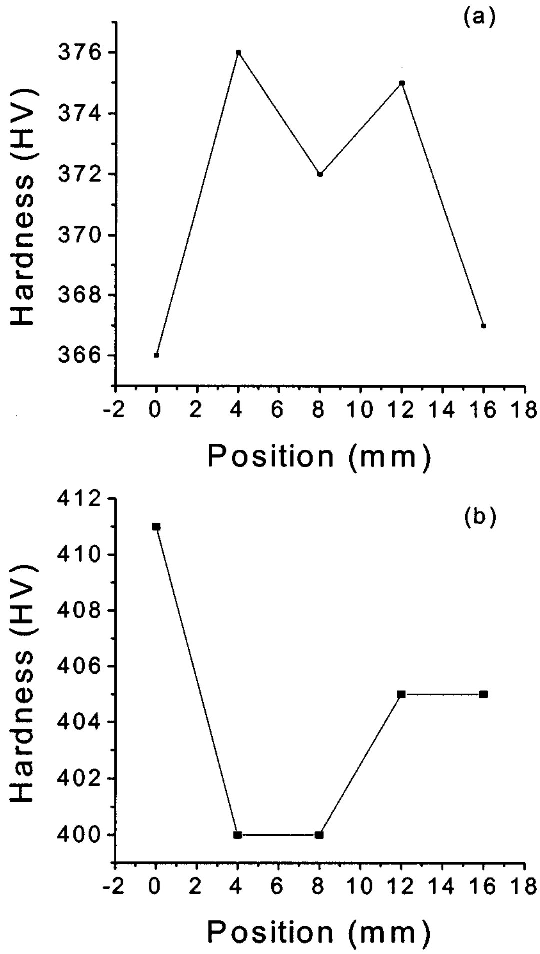

FIG. 9. Microhardness test results measured across the surface of the chosen sample of Fig. 8 with (a)—1 kg load; and (b) --300 g load.

only. Transversely inhomogeneous case hardened samples are not suitable candidates for the one-dimensional reconstruction as thermal diffusivity inversions can lead to inaccurate depth profile reconstructions.[7,8]

Hardness imaging criteria based on this work can be developed and used for determining if a sample is a suitable

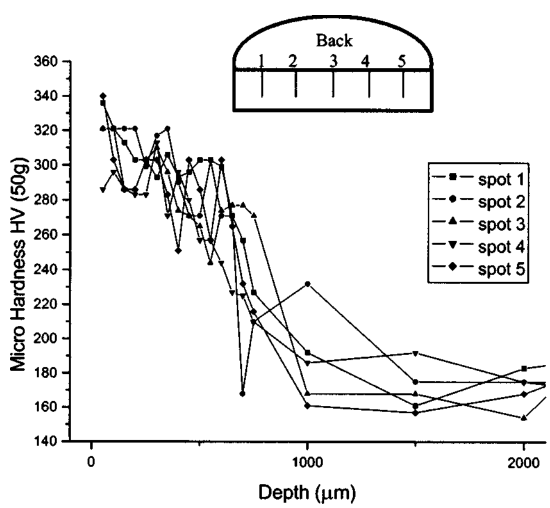

FIG. 10. Carbonitrided AISI 1018 sample 9 (0.04 in. case depth) back surface hardness depth profile with seven different measured spots.

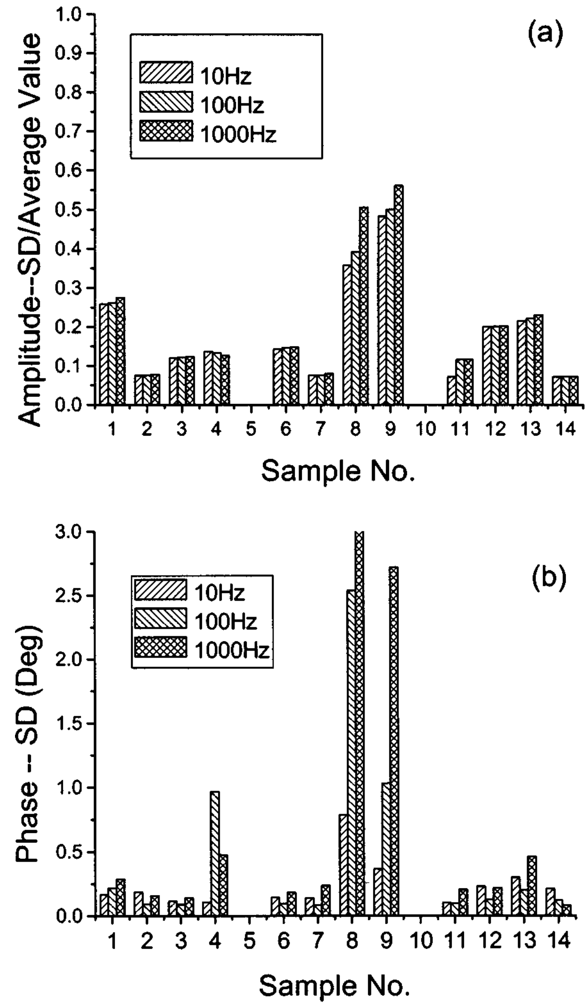

FIG. 11. Histogram of (a) amplitude and (b) phase line scans of a group of AISI 1018 steel samples.

candidate for reconstruction. Transverse line scans performed on each of the carbonitrided and quenched 1018 steel samples at three different frequencies, 10, 100, and 1000 Hz, were employed. At each frequency, the mean and variance of the data sets over the entire amplitude and phase lateral scan were calculated and the results plotted in a histogram. For amplitudes, the quotient of variance and mean at a particular frequency was plotted and for phase, the variance of the measurements taken at a particular frequency was plotted. The results of one such test are shown in Fig. 11. These histograms permitted the immediate identification of the least homogeneous samples. For example, from the histograms of Fig. 11, and application of the criterion that a steel sample is mechanically homogeneous if PTR phase variations across a surface scan are less than 0.5°, it is obvious that samples 8 and 9 are far less homogeneous than other samples of the group and thus should not be used in thermal diffusivity reconstructions. By using this line scan method of rejecting the less transversely homogeneous samples, a significant improvement in depth profilometric data inversion has been achieved.[10]

CONCLUSIONS

We have shown that industrial case hardened steel samples often contain thermophysical and mechanical transverse inhomogeneities which are well correlated. By scanning the surface using photothermal radiometry, an image of the mean thermal diffusivity down to a depth fixed by the thermal diffusion length of carbonitrided or otherwise hardened samples can be produced. Phase imaging of hardness inhomogeneities can be used effectively as a criterion for strongly inhomogeneous steels to be excluded from thermal-wave depth-profilometric reconstruction of thermal diffusivity or conductivity. A simple method has been proposed as a quantitative criterion to determine which samples should be chosen or rejected for such thermal-wave reconstructions. Subsequent satisfactory performance or failure in the field of such inhomogeneously hardened parts remains an open question.

ACKNOWLEDGMENT

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