ULTRASONIC SENSOR FOR CONTROL OF SURFACE MODIFICATION

B.J. Elkind, M. Rosen
Center for Nondestructive Evaluation
Materials Science and Engineering Department
The Johns Hopkins University
Baltimore, Maryland 21218

H.N.G. Wadley
National Bureau of Standards
Gaithersburg, Maryland 20899

Abstract

A nondestructive method is presented for the determination of depth and hardness of modified surface layers. This method is based upon the differences in the elastic constants and density between a modified surface layer and the substrate beneath it. These parameters can be conveniently observed as variations in velocity with changes in frequency of Rayleigh surface waves. As a test of this technique, studies were conducted on an AISI 1053 plain carbon steel that has been subjected to electron-beam irradiation. The electron-beam treatment allowed for the creation of thin, microstructurally-modified surface layers. The (frequency-independent) Rayleigh surface wave velocity of homogeneous pearlitic and martensitic samples of this steel were 3002 m/s and 2960 m/s, respectively. The lower velocities of the martensite resulted from a softening of the elastic constants. On samples with an approximate 1 mm rapidly solidified, martensitic surface layer on a pearlitic substrate, the Rayleigh velocity varied from 2984 m/s at low frequency (deep penetration into the substrate) to 2960 m/s at high frequency (penetration confined only to the surface layer). A rapid increase in velocity occurred as the depth of penetration extended beyond the depth of the modified surface layer and an empirical method for determining penetration depth (hence, layer thickness) to within 10%.

INTRODUCTION

Microcrystalline modified surface layers may possess enhanced resistance to erosion, wear and corrosion. These beneficial attributes are important in a diversity of high technology applications where material performance has been the primary limitation. If this material's processing approach is to be utilized in practice, it is important to control spatial nonuniformities (of microstructure and chemistry), porosity, cracking, depth of modification and surface topology. Microstructural and chemical heterogeneity are believed to be
caused by uncontrolled variations in process variables; principally temperature gradient, solidification rate, cooling rate, and by convective mixing in the melt. Microstructural inhomogeneities may also be caused by the annealing of already processed material by subsequent melt passes in adjacent material.

The formation of amorphous surface layers or the layer glazing process, as it is sometimes referred to, has been demonstrated to be technically feasible, but its widespread application is limited by two problems: the difficulty of producing deep (>20 μm) amorphous layers and the elimination of cracking. The former problem arises either from the reduction of temperature gradient for deeply melted layers, which causes a reduction of solidification velocity to a value below the threshold for glass formation[1] or from the occurrence of amorphous to crystalline transitions in the heat affected zones of previously vitrified material during sequential melting passes in adjacent material. The cracking problem, on the other hand, arises because of the large tensile residual stresses that are produced by solidification shrinkage strains.

The work reported here concerns development of an ultrasonic technique to characterize the depth of surface modified microcrystalline layers and their hardness. Clearly, if the potential of surface modification with directed high energy sources is to be fully realized, techniques (or sensors) need to be developed to measure, in process, key process variables (those controlling microstructure) and to detect cracking and other deleterious conditions. With such a capability it may then be possible to use automated process control[2] as a means for reliably producing uniformly high quality material. Previous research[3] has shown the potential of acoustic emission for detecting cracking during surface modification.

The basis of the technique stems from the difference in ultrasonic velocity between the layer and substrate. In this study, the technique has been applied to the characterization of a surface modified layer on steel substrate. Since the physical basis of the technique is the difference in ultrasonic velocity of substrate and layer, it should be noted that it is actually more pronounced if the surface layer is amorphous, because the difference in elastic constants, and thus ultrasonic velocity, of the (crystalline) substrate and the (amorphous) layer is larger. Consequently, the microcrystalline layer study is in some respects a worst case test of the approach.

When applied to steels, electron beam surface modification has been observed to cause substantial improvements in hardness due to the formation of martensite/bainite and carbide precipitate refinement.[4] Systematic investigations have shown that the hardness and depth of modification are controlled by scanning rate.[5-7] Thus, variation of the scan velocity could provide a convenient potential method for the control of hardness and melt depth, provided they can be measured in-process. The use of ultrasonic surface acoustic waves (Rayleigh waves) has been investigated here for the characterization of the depth and hardness of electron beam modified surface layers.
For NDE surface layer characterization, the technique utilized was the ultrasonic generation of Rayleigh surface waves. These waves can be induced in a material by means of piezoelectric generation and detection with standard ultrasonic transducer. The surface wave energy diminishes rapidly with increasing depth into the bulk of a material. The rate of decrease in amplitude is, however, dependent upon wavelength; at a depth of approximately one wavelength, the particle amplitude is only about 20% (and the particle energy is only 4%) of that at the surface. Therefore, this waveform represents a two-dimensional wave that attenuates as 1/r (r is the distance from point source). In addition, Rayleigh wave propagation in a uniform material is nondispersive in that Rayleigh velocity is independent of frequency. The velocity is dependent only upon density and elastic constants in homogeneous media.

The velocity ($V_R$) of Rayleigh surface waves can be expressed in terms of a constant ($\gamma$), and the shear wave velocity and Poisson’s ratio ($\nu$):

$$\gamma = \frac{V_R}{V_t} = \frac{0.87 + 1.12\nu}{1 + \nu}$$

Eq. #1

Rayleigh wave fronts are elliptical in nature and travel along the direction of propagation in a planar fashion and orthogonal to the surface. This wave motion is illustrative of the fact that the Rayleigh wave is a combination of vertically polarized shear (SV) waves and longitudinal waves. The major axes of these ellipses depict the vertical particle displacements (SV motion) of the Rayleigh waves, whereas the minor axes represent the horizontal (or longitudinal) displacements. For surface layer analysis, the orthogonal component of the Rayleigh wave motion is of principal interest and the effective depth of penetration of the Rayleigh surface wave may be assumed to be approximately one wavelength ($\lambda_R$).

The penetration depth of the Rayleigh waves may be varied by changing the frequency of the waves of constant velocity (in an homogeneous medium) according to the relation: $V = f\lambda$ where $V_R$ - Rayleigh surface wave velocity (phase), $f$ is the Rayleigh wave frequency and $\lambda$ is the Rayleigh wavelength, which can be assumed to be equal to the penetration depth. Therefore, the measurement of velocity dispersion could potentially provide rapid, non-destructive method for selectively probing subsurface properties[8-10].

**EXPERIMENTAL**

**Specimen Preparation and Characterization**

Several 7.6cm x 2.5cm x 0.6 cm slabs of AISI 1053 (0.53% carbon) steel were austenitized at 1025°C for 45 minutes. One of the samples was subsequently quenched in an ice-brine to produce a bulk martensite. The remaining samples were furnace cooled to to 650°C, allowed to isothermally transform for 15 minutes and air-cooled to room temperature. This resulted in a pearlitic microstructure. The specimens were eventually subjected to electron-beam surface
heat-treatments. The electron-beam glazing technique employs a high intensity/energy collimated beam of electrons. The directed energy beam strikes the surface of metal or alloy, causing the subsequent heat-treatment (or melting) of a surface layer. High cooling rates on the order of $10^6$ K/s are obtained by self-substrate quenching. The parameters that dictate the effectiveness of this process are the following: beam energy, beam intensity, beam shape, and scan rate.

After completion of the preliminary characterization by means of optical metallography and mechanical testing, each of the samples was subjected to an identical surface modification process. A 25 KV accelerating voltage was employed to accelerate a 2.5 mm diameter beam of electrons (emitted from a tantalum filament) to the specimen surface. The beam current was 9 milli-amps. The beam power was, therefore, 225 watts. The scan rate of the electron-beam across the length of each specimen was 0.3 cm/s. After each scan of the electron-beam, the beam was translated 0.0254 cm across the width of the specimen, so as to microstructurally modify the entire surface. After the initial heat treatments of the specimens to obtain bulk pearlitic and martensitic specimens, several preliminary measurements were performed. Rockwell macrohardness tests were conducted and the densities of the specimens were measured. In addition, standard metallography and microstructural analyses were carried out. Longitudinal and shear ultrasonic bulk wave velocity measurements were obtained on these samples, utilizing the pulse-echo overlap technique. Elastic moduli were calculated from the ultrasonic data.

Ultrasonic Rayleigh Wave Characterization

In the present investigation, the generation of Rayleigh waves is obtained by means of a specially designed and constructed mode-conversion device. An illustration of this device appears in Figure 1. The wedge device consists of two machined 2024 aluminum wedges that are placed in an aluminum sliding mount. Each wedge has an active surface area in contact with the specimen of approximately 2.54 cm long by 700 μm wide (w). A piezoelectric transducer is mounted inside each wedge; the wedges are machined down to knife-edge and the sample is approximately 60°. The generation and detection of the Rayleigh waves are accomplished by conversion of the generated longitudinal wave and by subsequent reconversion of the detected wave.

Rayleigh wave frequencies from 2.2 to 15.5 MHz were used to characterize electron-beam treated samples. The minimum value of frequency was determined by the need to have at least a half-wavelength equal to (or less than) the width of the wedge i.e.,

$$\frac{3002 \text{ m/s}}{1400 \text{ μm}} = 2.1 \text{ MHz} \quad \text{Eq. \#2}$$
At the lower frequency limit, waveguide effects predominate, thereby prohibiting low frequency mode conversion coupling of longitudinal waves into Rayleigh waves to the sample. The observed frequency minimum of 2.2 MHz correlates well with the theoretically predicted frequency minimum of 2.1 MHz. At high frequencies, difficulties arise from enhanced attenuation, which increases with frequency. A likely source of this is grain boundary scattering [11].

RESULTS

Homogeneous Samples

The results of preliminary characterization of homogeneous pearlitic and martensitic microstructure specimens are shown in Table 1. Noteworthy, in Table 1 is the observation that both the longitudinal and shear wave velocities of pearlite are greater than those of martensite, an observation that has been reported by others. [12]

For isotropic linear elastic materials, the density and wavespeeds can be used to determine Young’s modulus, the bulk and shear moduli and Poisson’s ratio. [9] Furthermore, it is possible to predict the Rayleigh wave velocity, $V_R$, using the relation expressed in Eq. 1. These physical quantities are presented in Table 2.
Table 1
MEASURED PHYSICAL PROPERTIES OF HOMOGENEOUS AISI 1053 SAMPLES

<table>
<thead>
<tr>
<th>PHYSICAL QUANTITY</th>
<th>PEARLITE</th>
<th>MARTENSITE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>7796 Kg.m$^{-3}$</td>
<td>7779 Kg.m$^{-3}$</td>
</tr>
<tr>
<td>Shear Wave Velocity</td>
<td>3239 m.s$^{-1}$</td>
<td>3167 m.s$^{-1}$</td>
</tr>
<tr>
<td>Longitudinal Wave Velocity</td>
<td>6011 m.s$^{-1}$</td>
<td>5945 m.s$^{-1}$</td>
</tr>
<tr>
<td>Rockwell Macro-Hardness</td>
<td>$R_B = 91.5$</td>
<td>$R_C = 57.0$</td>
</tr>
</tbody>
</table>

Table 2
DEDUCED PHYSICAL PROPERTIES OF HOMOGENEOUS AISI 1053 SAMPLES

<table>
<thead>
<tr>
<th>PHYSICAL QUANTITY</th>
<th>PEARLITE</th>
<th>MARTENSITE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young’s Modulus (E)</td>
<td>211.9 GPa</td>
<td>203.1 GPa</td>
</tr>
<tr>
<td>Bulk Modulus (K)</td>
<td>172.7 GPa</td>
<td>170.9 GPa</td>
</tr>
<tr>
<td>Shear Modulus (G)</td>
<td>81.78 GPa</td>
<td>78.00 GPa</td>
</tr>
<tr>
<td>Poisson’s Ratio($\nu$)</td>
<td>0.295</td>
<td>0.301</td>
</tr>
<tr>
<td>Rayleigh wave velocity ($V_R$)</td>
<td>3002 m.s$^{-1}$</td>
<td>2939 m.s$^{-1}$</td>
</tr>
</tbody>
</table>

Microstructure of Surface Modified Material

The surface-modified samples (sectioned perpendicular to the electron beam pass direction), polished and etched in 2% nital to reveal the microstructures produced and to facilitate microhardness measurements. Figure 2 shows, at low magnification, a view of a region about midway across the width of AISI 1053 modified material shown together with a microhardness depth profile. Starting at the bottom, it can be seen that the substrate had a pearlite microstructure with a grain size of 25 $\mu$m, (Figure 2), the peak temperature experienced during modification increases. This at first results in dissolution of cementite, then a transformation to austenite and finally melting during the heating part of the thermal cycle. Rapid cooling from these states then produces a wide spectrum of microstructures that are vertically separated. Subsequent melt passes further complicate the microstructures due to tempering in the heat affected zone. Results of microhardness and microstructural analysis of modified AISI 1053 are summarized in Table #3.
Figure 2. A narrow region of the sample revealing the vertical separation of micro-structure (2% Nital etch) together with the micro-hardness variation with depth.
Table 3

PROPERTIES OF SURFACE MODIFIED AISI 1053 ALLOY

<table>
<thead>
<tr>
<th>REGION</th>
<th>THICKNESS</th>
<th>MICROSTRUCTURE</th>
<th>HARDNESS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melt Layer</td>
<td>0.330 mm</td>
<td>Tempered Upper Bainite</td>
<td>$R_e=22-29$</td>
</tr>
<tr>
<td>Circular Layer</td>
<td>0.435 mm</td>
<td>Bainite</td>
<td>$R_c=27-30$</td>
</tr>
<tr>
<td>Transition Zone</td>
<td>0.300 mm</td>
<td>Pearlite/Spheroidal Carbides</td>
<td>$R_c=22-29$</td>
</tr>
<tr>
<td>Substrate</td>
<td>5.2 mm</td>
<td>Pearlite</td>
<td>$R_b=94.0$</td>
</tr>
</tbody>
</table>

Rayleigh Wave Measurements

Figure 3 shows a plot of the measured Rayleigh wave velocity as a function of wavelength (penetration depth) for the surface modified layer and the unmodified pearlitic substrate as obtained from the unmodified reverse side of the sample. It can be seen that the substrate exhibits a constant velocity independent of wavelength with an average value of 3001 m/s in excellent agreement with the predicted value (in Table 2) from bulk wave measurements. The absence of dispersion from a homogeneous sample indicates that the experimental approach does

![Rayleigh Wave Measurement Graph](image_url)

Figure 3. VARIATION OF THE RAYLEIGH WAVE VELOCITY WITH WAVELENGTH (PENETRATION DEPTH) FOR AISI 1053 STEEL.
not introduce any significant velocity measurement errors. The velocity measured on the surface modified layer exhibits significant dispersion. At high frequencies (shallow penetration depths) a limiting velocity of 2960 m/s is achieved. This represents the velocity of the layer and is 20 m/s greater than the value predicted from bulk wave measurements on a homogeneous martensite sample. This difference is most probably due to the bainite microstructure (Figure #4) of the layers since bainites are well known to have elastic properties intermediate between those of pearlite and martensite. [12] We see in Figure 3 that as the wavelength increased the velocity began to approach the value of pearlite consistent with an enhanced contribution of the substrate to the elastic constants sampled by the wave.

Figure 4. BAINITIC MICROSTRUCTURE INDICATIVE OF THE MODIFIED SURFACE LAYER IN AISI 1053 (2% NITAL ETCH @ 1250 x MAG.).

DISCUSSION

Rayleigh waves propagating over the surface of electron-beam modified surfaces, sample only the modified (martensitic) layer at high frequencies (since the wave does not penetrate the layer) and some combination of the layer and substrate (pearlitic) at lower frequencies. In the high frequency region, the velocity is found to be approximately frequency independent, consistent with wave propagation (theory) in an approximately uniform, non-dispersive medium. Then the limiting velocity can be viewed as a characteristic of the layer microstructure and not its depth. The velocity for AISI 1053 surface layers is intermediate between that anticipated for martensite and pearlite, and is consistent with the observed bainite state.
There thus appears considerable merit to the use of this asymptomatic value to characterize layer hardness.

Rayleigh waves with greater penetration depths yielded velocities that were intermediate between those of the substrate and the layer. Provided the substrate is much thicker than the layer, then when the wavelength (penetration) is very much greater than the layer thickness, the velocity approaches the Rayleigh velocity for pearlite because contributions from the layer become increasingly small. This limit was not reached in these samples due to their finite thickness, but sufficient data for a depth determination were obtained nevertheless.

To determine the layer thickness, the region of interest is the intermediate velocity region, where the velocity results from the simultaneous sampling of several different microstructures. For example, in AISI 1053 at a frequency of 4.2 MHz (penetration depth = 705 m), the Rayleigh velocity was 2960 m/s; however at 2.2 MHz (penetration depth = 1357 m), the apparent Rayleigh velocity was 2984 m/s. The velocity thus depends upon both the characteristic velocities of the layer and substrate and the layer depth.

The inverse problem of deducing the layer properties from velocity dispersion data is complex and is the object of ongoing study. Here, we use a very simple analysis that gives results of reasonable accuracy. For the case where the wave penetrates the surface modified layer, we may, to a first approximation, assume the apparent velocity to be determined by the layer thickness: wavelength ratio according to a law of mixtures:

\[ V = XV_L + (1-X)V_S \quad \text{Eq. #3} \]

where \( V_L \) = the velocity of the layer

\( V_S \) = the velocity of the substrate

\( x = x_L / \lambda \), the ratio of layer thickness to wavelength

(n.b. we define \( x = 1 \) for \( \lambda X_L \))

In the long wavelength limit, \( x \) tends to zero and the apparent velocity is \( V_S \), while for \( \lambda X_L \), the velocity becomes that of the layer, \( V_L \).

To determine the layer depth we rearrange the equation above:

\[ X_L = \frac{(V-V_S) \cdot \lambda}{V_L - V_S} \quad \text{Eq. #4} \]

Substituting data from Figure 3 into this expression yields a value for a modified layer depth of 600-800 \( \mu \)m. The variability depends upon the precise value of wavelength taken. Metallography for AISI 1053 indicated that the combined circular and melt depth was 850 \( \mu \)m, in reasonable agreement to that deduced bearing in mind the simplicity of a law of mixture model.
A more complete analysis incorporating exponential weighting of the Rayleigh wave particle displacement, might give a more exact inversion, and thus deeper insight, into the functional behavior of the modulus with depth. However, for the present purpose, this simple approach appears sufficient, at least for the AISI 1053 alloy. Indeed, an even simpler depth determination criterion such as the penetration depth at which the velocity curve increases 2 m.s⁻¹ above the short wavelength velocity limit predicts the metallographically deduced depths of hardening for both AISI 1053 and 1044 alloys that have been subjected to surface modification to within +25μm.

The Rayleigh wave technique has proved, therefore, to be an accurate and reproducible technique for determining the depth of hardening of surface-modified steels. Since it is nondestructive, and methodologies which would not interfere with processing are emerging, it can potentially be used for on-line quality control purposes. If account is taken of the effect of sample temperature fluctuations on the velocity, it could serve the role of a process control sensor for feedback control of depth of modification and hardness, particularly if emerging noncontact measurement methodologies are utilized.

SUMMARY

Samples of pearlitic plain carbon steels were subjected to an electron-beam glazing process resulting in a (case-hardened) microstructurally modified surface layer.

Hardness and ultrasonic velocity data indicated that the modified surface layers possessed a higher hardness but a lower velocity than the substrate materials. This decrease in velocity may be a good indication of the level of hardness attained in the modified layer.

Dispersion of the Rayleigh wave velocity can be utilized to make a nondestructive estimate of the depth of the microstructurally modified surface layer.

ACKNOWLEDGEMENTS

We are grateful to Dr. R. Schaefer for the assistance and advice with electron beam surface modification, to C. Brady for help with metallography and to the Defense Advanced Research Projects Agency for funding of this work under DARPA Order Number 4275 (Major S. Wax, Program Monitor).

REFERENCES


6. Ibid, p. 343


