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Laser-Acoustic Characterization of Coatings

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Abstract

Non-destructive, contactless and robust characterization of coatings is an ongoing and challenging task in the development of measurement technologies. The determination of elastic material parameters of coating and substrate can serve as a convenient method for the characterization of coatings. In our measurement system, elastic material parameters were derived from the dispersion of surface acoustic waves. The ultrasound waves were excited in the thermoelastic regime using a pulsed laser and were measured in the near-field of the excitation region using a heterodyne Mach-Zehnder interferometer. The dispersion of the surface waves was calculated from the measured time-domain traces using the phase spectral analysis method. The dispersion curves were simulated using a single-layer model. The simulation of the dispersion of Rayleigh waves on a layered half-space was then fitted to the measured dispersion curves using a least-squares algorithm. With this measurement method it was possible to measure the average thickness and thickness distribution of polymer coated aluminum specimen with high accuracy in a scanning manner.

Key words: Laser-Based Ultrasound, Surface Acoustic Waves, Coatings

Introduction

Laser-Based Ultrasound (LBU) is a common tool for the inspection of coatings on a solid half-space [1]. Recent approaches often featured a contacting measurement method [2] or focused on the characterization of thin films with rather low acoustical attenuation [3][4].

The theory of Rayleigh wave propagation on both an isotropic half-space [5] and on a layered half-space [6] is well established. Computer programs can be used to efficiently calculate and fit a theoretical phase velocity dispersion curve to a measured dispersion curve.

The characterization of polymer coatings in the range of 5 – 20 µm on untreated, industrial-grade aluminum remains a challenging task. The attenuation of surface acoustic waves (SAW) on polymer coatings is very high. This results in a limited frequency range that can be considered for analysis. In order to overcome this challenge, several measures were considered in the measurement routine.

First, excitation laser power was increased to a level just beneath the ablation threshold of the coatings. Second, measurements were carried out in the near-field close to the source, in order to avoid diffraction phenomena and to minimize acoustic attenuation. Third, a sufficient distance from source to receiver of some millimeters has to be maintained in order to minimize uncertainties in the phase velocity [7].

Theory

Polymer coatings on a half-space introduce a dispersive behavior on otherwise non-dispersive Rayleigh surface waves. In order to model this dispersive behavior, the dispersion curves of a one-layer model were calculated using the derivation of Tiersten [6].

Using the Helmholtz decomposition, the wave equations can be written in terms of longitudinal potentials Φ and transversal potentials Ψ, where a comma followed by a number is the derivation with respect to the corresponding space coordinate:

$$\phi_{,11} + \phi_{,22} + k_l^2 \phi = 0,$$

$$\psi_{,11} + \psi_{,22} + k_t^2 \psi = 0. \qquad (1)$$

Using relations

$$\eta = \sqrt{k^2 - k_p^2},$$

$$\beta = \sqrt{k^2 - k_s^2}\,, \qquad (2)$$

with k = wavenumber of the Rayleigh wave and $k_{p/s}$ = wavenumber of longitudinal and transverse wave, the solutions of Rayleigh-like surface waves in the half-space and layer are:

$$\phi^2 = a_2 \cdot e^{-\eta_2 x_2} \cdot e^{ikx_1}$$

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$$\psi^2 = c_2 \cdot e^{-\beta_2 x_2} \cdot e^{ikx_1}$$
$$\phi^1 = (a_1 \cdot e^{-\eta_1 x_2} + b_1 \cdot e^{\eta_1 x_2}) \cdot e^{ikx_1}$$
$$\psi^1 = (c_1 \cdot e^{-\beta_1 x_2} + d_1 \cdot e^{\beta_1 x_2}) \cdot e^{ikx_1}, \quad (3)$$

where the superscript 2 denotes the solution in the half-space and superscript 1 denotes the layer. Coefficients a_1 … c_2 are unknowns and have to be determined.

The potentials can be used to calculate the displacements:

$$u_1 = \phi_{,1} + \psi_{,2}$$
$$u_2 = \phi_{,2} + \psi_{,1}, \quad (4)$$

with u_1 = horizontal displacement and u_2 = vertical displacement.

From the displacements, strains

$$\epsilon_{ij} = \frac{1}{2}\left(u_{i,j} + u_{j,i}\right) \quad (5)$$

and, subsequently, stresses

$$\sigma_{ij} = \lambda(\mathrm{u}_{1,1} + \mathrm{u}_{2,2})\delta_{ij} + 2\mu\epsilon_{ij} \quad (6)$$

can be calculated. In Eq. (6), λ and μ are Lamé-constants.

In case of a Rayleigh surface wave, the stress - free boundary conditions

$$\sigma_{i2}^1 = 0 \; @ \; x_2 = 0 \quad (7)$$

can be applied at the surface and continuing stresses and displacements can be applied at the interface between half-space and layer:

$$\sigma_{i2}^1 = \sigma_{i2}^2 \; @ \; x_2 = h$$
$$u_i^1 = u_i^2 \; @ \; x_2 = h. \quad (8)$$

With help of the boundary conditions, a system of equations for the unknown coefficients can be formed:

$$\vec{D} \cdot \begin{pmatrix} a_2 \\ c_2 \\ a_1 \\ b_1 \\ c_1 \\ d_1 \end{pmatrix} = \vec{0}. \quad (9)$$

The coefficient matrix $\vec{D}$ is of the size 6x6. Of this system of equations there only exist non-trivial solutions for singular values of the coefficient determinant $|\vec{D}|$.

In order to find the singular values of $|\vec{D}|$, and, thus, the corresponding Rayleigh wave velocity, a starting frequency f_0 is chosen. At this frequency, $|\vec{D}|$ is calculated using velocities in a range from c_0 to c_{end}. If there is a change in the sign of $|\vec{D}|$, a singular value was passed. A bisection algorithm is then used to find the exact value of c_R within a specified error bound ε. Thereafter the frequency is increased by a value of Δf and $|\vec{D}|$ is computed for velocities c_0 … c_{end} again. After velocities at two frequencies are found, a linear extrapolation algorithm is used in order to predict the subsequent velocity. The next solution of the Rayleigh mode is searched after in direct vicinity of the predicted value. This way, only a single Rayleigh wave mode can be traced. However, only the fundamental Rayleigh mode is considered in the experiments and Sezawa modes are neglected.

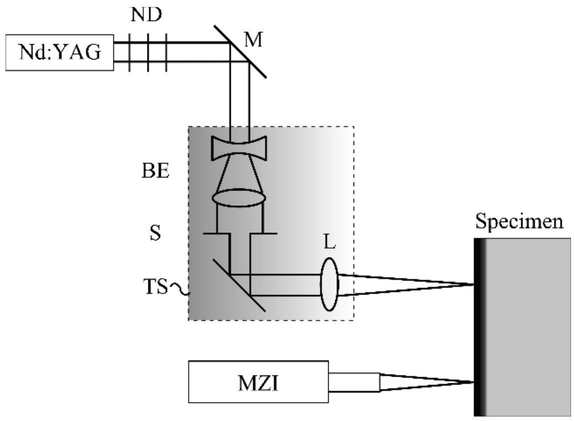

Fig. 1 Schematic of the experimental setup. ND: neutral density filters, M: mirror, BE: beam expander, S: slit aperture, TS: translation stage, MZI: heterodyne Mach-Zehnder interferometer, L: cylindrical lens.

Experiment

The basic measurement setup was described earlier [8]. A schematic of the setup is displayed in Fig. 1. A Nd:YAG laser with pulse duration of 3 ns and pulse energy of 160 mJ was used to generate the ultrasound waves. The beam diameter of the laser beam was 5 mm. The pulse energy was attenuated with neutral density filters to a degree where no ablation was observable. A cylindrical lens was used to focus the beam to a thin line onto the surface of the specimen. A heterodyne Mach-Zehnder interferometer was used to detect the surface displacement of the SAW.

However, the setup was improved in a way that near-field displacements could be detected. Before the laser beam was focused by the cylindrical lens, it was expanded with a Galilean beam expander. In order to avoid spherical aberration, a slit was used to block off-center rays on the cylindrical lens. Due to the beam expander, the line length is about 15 mm. The distance from source to detection was between 4 mm to 14 mm, where there was a plane wavefront of SAW in the near field of the source.

Because of beam expansion, the slit aperture and losses at the optical surfaces, the energy density in the focal plane of the cylindrical lens drastically decreased. To compensate for this losses, the pulse energy was increased by removing neutral density filters.

The phase-spectral analysis (PSA) method [9] was used in order to calculate the experimental dispersion relation from displacement measurements. The PSA is based on the formula

$$c(f) = \frac{2\pi f \cdot (x_2 - x_1)}{\Phi_2(f) - \Phi_1(f)}, \quad (10)$$

where $x_{1,2}$ = different distances from source to receiver, $\Phi_{1,2}$ = phase at distance 1 and 2. Different positions $x_{1,2}$ were adjusted precisely with a linear stage.

The specimen consisted of a cylindrical aluminum disk with an untreated surface. A drop of the polymer solution was applied to the center of the surface and it was subsequently applied to a spin coater. Spin coating speeds of 50 rpm, 70 rpm and 90 rpm were used, resulting in layer thicknesses between 5 µm and 20 µm.

The layer thickness distribution was additionally measured with an inductive caliper Mahr P2004 and a Millimar C1216 length measurement instrument. The repeatability of the caliper is 0.1 µm. In order to measure the layer thickness distribution, the specimen were mounted on top of a linear stage and scanned beneath the caliper.

Results

In Fig. 2 a caliper measurement of a spin coated specimen is shown. As can be seen from the measurement curve, the thickness distribution is not uniform. Any measurement of SAW along a line of this profile thus results in an average value.

In Fig. 3 the measured dispersion curves of the raw surface of the cylindrical aluminum disk and

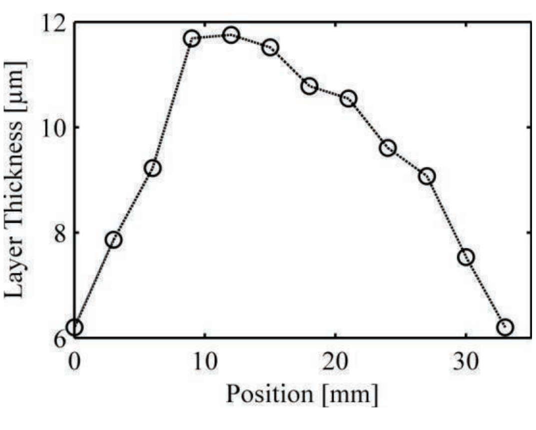

Fig. 2 Caliper measurement of the thickness distribution of a spin coated polymer coating on a cylindrical aluminum disk.

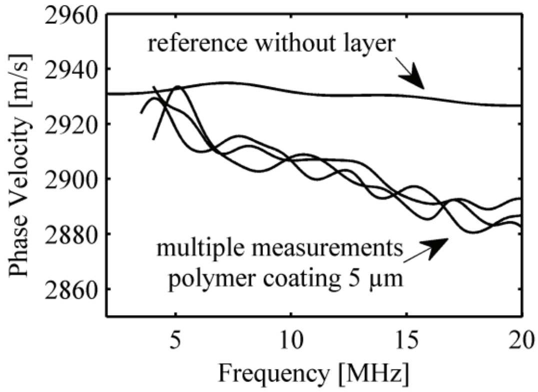

Fig. 3 Comparison between measured dispersion curves of uncoated and coated surfaces.

multiple measurements of the layer are shown. Measurements of the layer were carried out very close to each other, with a resulting average thickness similar for each measurement. While the Rayleigh wave phase velocity of the uncoated surface remains nearly constant in the whole frequency band, the velocity of the Rayleigh-like waves in the layered system decreases with increasing frequency. This is because the penetration depth of the Rayleigh wave decreases with increasing frequency. Therefore, high frequency waves are more sensitive to the thin coating with slow velocity than waves at lower frequencies. As can be seen from the repeated measurements in Fig. 3, the measurement uncertainty is about $\pm 5\,\frac{m}{s}$ in the frequency range under consideration.

In Fig. 4 measurements with different average layer thicknesses are shown. Effects of the underlying aluminum surface have been compensated for and the change in velocity is shown in percent. From the best-fitting simulation of the Rayleigh-like waves, the layer thickness was found. Lamé constants of the layer have been found by first measuring the average thickness of a layer with the caliper and

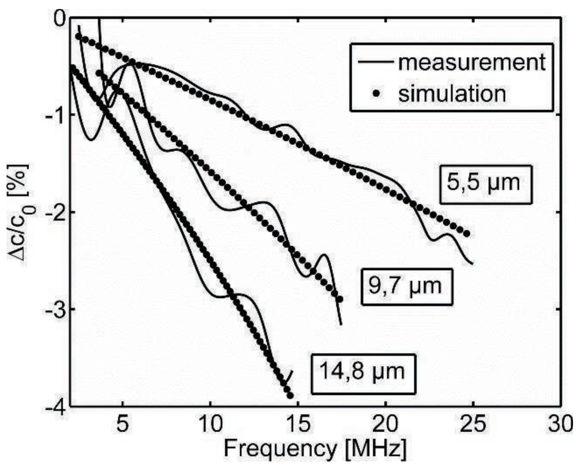

Fig. 4 Dispersion curves of Rayleigh waves on layers with different average thickness.

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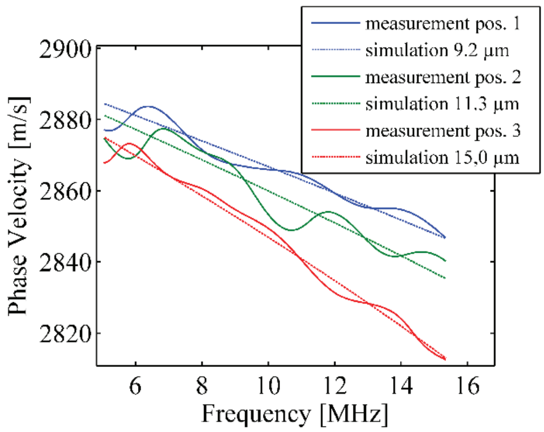

Fig. 5 Measured and best-fitting simulated dispersion curves for positions 1, 2 and 3.

then fitting the simulated dispersion curve to the measured dispersion curve by varying the material constants and keeping the layer thickness constant. The material constants found for this type of polymer layer are $\lambda = 10.43\ GPa, \mu = 2.607\ GPa, \rho = 1.2\frac{g}{cm^3}$.

The thickness distribution of a spin coated specimen was measured with a caliper at 170 positions. From the measured distribution, average values were calculated in the regions in which SAW measurements were carried out. In total, five parallel lines with a spacing of 5 mm across a specimen were measured using the aforementioned SAW method. Dispersion curves of three of them are shown in Fig. 5. It can be seen that the average thickness differs drastically between the edge of the specimen ("pos. 1") and the central area of the specimen ("pos. 3").

In Fig. 6 a comparison between the calculated average values from the caliper measurement and the SAW thickness-measurement is displayed. The error bars result from the deviation in thickness values from the caliper measurement along a scan line. Since the

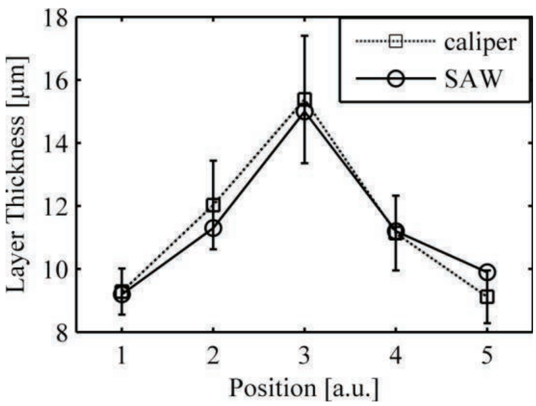

Fig. 6 Average layer thickness of a spin coated polymer coating on a cylindrical aluminum disk at different positions.

thickness distribution is highly inhomogeneous, the error bars indicate the inhomogeneity of the layer and are much greater than the uncertainty of the caliper instrument. However, there is a very good agreement between caliper and SAW measurements.

Conclusion

It could successfully be shown that the method of laser-based surface acoustic wave measurements can be adopted to the determination of properties of highly inhomogeneous and strongly absorbing polymer coatings.

Measurements were carried out in the near-field of the laser source with a heterodyne Mach-Zehnder interferometer. The laser power was attenuated in order not to damage the layer and guarantee a thermoelastic excitation mechanism.

The SAW dispersion was theoretically calculated using a one-layer model. For the determination of material constants of the polymer coatings, the simulation was fitted to a measurement at known layer thickness.

Comparison between the average thickness distribution of a spin coated specimen measured with a caliper and subsequently measured with the SAW device shows great consistency. The average thickness profile can be reconstructed with the SAW measurements without altering the material parameters in the SAW simulation.

In current profile measurements, the specimen was translated in order to move to different positions for the SAW measurements, resulting in an average layer thickness profile. In future experiments a more exact reconstruction of the profile could be achieved using tomographic algorithms like radon transformation instead.

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