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Influence of a superficial field of residual stress on the propagation of surface waves—Applied to the estimation of the depth of the superficial stressed zone

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In this study, we were interested in the dispersion of surface waves caused by the presence of a micrometric field of residual stress on the surface of an amorphous medium. We have shown that in relation to surface waves, a stressed structure like this is comparable to a layer on substrate type structure. The design and implementation of SAW-IDT MEMS sensors enabled quasi-monochromatic Rayleigh-type surface waves to be generated and the dispersion phenomenon to be studied over a wide range of frequencies for different superficial fields of residual stress. The thicknesses of the stressed cortical zones were estimated with good accuracy using an inverse method. © 2012 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4768434>]

The study and control of residual mechanical stress are of increasing importance in numerous fields such as microelectronics and materials with property gradients with a view to meeting new functional requirements. The effects of these stresses can be harmful in certain applications (breakdown phenomenon)¹ and sometimes beneficial like, for example, to improve the transport properties in silicon² or to mechanically reinforce glass.³ In this study, the influence of the superficial field of stress, of which the depth is in the order of tens of micrometers, on the propagation of surface waves was studied theoretically and experimentally on chemically tempered amorphous solids. Based on the theory of acoustoelasticity, this study shows the influence of such a field of stress on the propagation of surface waves and in particular highlights dispersion phenomena. But, in order to characterize this dispersion, it was necessary to generate and detect surface waves over a large frequency range. In this study, we developed and used SAW-IDT MEMS. This original solution is based on the development of an interdigital transducer to generate quasi-monochromatic surface waves and to obtain a rapid and accurate estimation of the phase velocity, key information in the characterization of superficial stress. Moreover, HF surface waves could be generated over a broad frequency range up to 60 MHz using SAW-IDT MEMS.⁴

Acoustoelasticity, i.e., the dependence of the acoustic wave velocity on the stress in an elastic material, has been studied for more than 40 years.^{5,6} Acoustoelasticity is based on a continuum theory of small disturbances (ultrasonic waves) superimposed on an elastically stressed medium formulated by Cauchy in 1929. The general theory of finite deformation was presented by Murnaghan⁵ and later in a treatise by Truesdell and Toupin.⁷ A modern theory of acoustoelasticity has been presented introducing third-order elastic constants in the constitutive equation.^{6,8,9} This formalism requires the deformation energy to be developed up to 3rd order, making the theoretical developments very

cumbersome. Different developments^{6,10–15} have all shown that it is possible to introduce the notion of effective elastic constants (EEC), C_{IJKL}^a with: $C_{IJKL}^a = C_{IJKL} + \delta C_{IJKL}$, where C_{IJKL} corresponds to the second order elastic constants and δC_{IJKL} to the disturbance linked to the presence of an applied or residual stress. Through the introduction of EEC, it is possible to use a classic formalism (second order) in the case of stressed materials. A stressed material can be considered, via the EEC, as a stress free material presenting second order elastic constants different from the second order elastic constants of an unstressed material.¹⁶

In numerous cases, fields of stress appear during the processing of materials, with sudden variations in temperature often being the cause of residual stress. However, chemical and mechanical causes also exist. In the context of amorphous materials such as glass, the fragility of the material can be limited using surface reinforcement by chemical or thermal tempering. The principle of chemical tempering (CT) of glass consists in replacing the sodium ions by more “voluminous” potassium ions. This chemical reaction occurs by diffusion by immersing the glass in a solution of molten potassium nitrate. A rectangular stress profile, also called “U” profile, is thus obtained (Fig. 1). An important characteristic of this tempering is the introduction of a superficial field of stress on the surface.^{17–19}

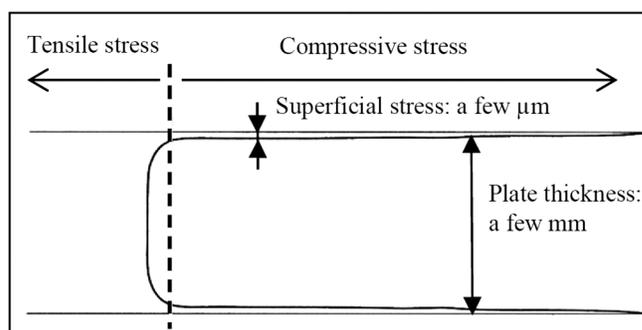


FIG. 1. Depth-wise stress profile of chemically tempered glass.

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This superficial stress is a compressive stress which can be in the order of several hundred MegaPascals and only affects a surface layer of which the thickness is in the order of a few tens of micrometers. Given the “U” (Fig. 1) profile of the field of stress, this chemically stressed structure can be considered as a structure composed of three uniformly stressed parts: first, two superficial zones (either side of the sheet) of micrometric thickness and under high compressive stress, and second, a central zone under low tensile stress. Consequently, given the description of stressed media and effective elastic constants, the chemically stressed glass sheets can be considered as structures made of a layer (one each side) with intrinsic properties different to those of the central substrate. The influence of a superficial field of residual stress on the propagation of surface waves on three chemical immersions of 3, 12, and 24 h was thus studied. Considering the surface wave frequencies chosen (5–60 MHz), the chemically stressed sheets of glass were considered here as “layer on semi-infinite substrate”-type structures.

When a SAW propagates on the surface of a material its energy is concentrated within a thickness of about one wavelength beneath the surface. When this wave propagates in a layer on substrate structure, the surface wave becomes dispersive.^{9,20,21} The phase and group velocities of this wave partly depend on the characteristics of the layer. In this study, the theoretical dispersion curves were obtained using a program based on a polynomial method allowing the specific acoustic modes of the structure to be determined.²² This method has been developed and validated in our laboratory specifically for the characterization of quantum semiconductor devices using acousto-opto-electronic interactions and for non-destructive testing. In order to calculate the phase velocities of the surface modes, the layer (compressive stress zone) and the substrate (tensile stress zone) were defined by their density and EEC in order to take into account the “perturbation” due to the presence of a residual stress profile. No contraction of indices was performed with the effective elastic constants, thus avoiding splitting that can occur using the contracted notations.¹⁶ The stress field here is, however, biaxial (plane stress) and isotropic in the plane.

The unordered structure of glass confers it total isotropy and high homogeneity on a macroscopic scale. At ultrasound scale, glass appears homogenous and isotropic. The second- and third-order constants for the glass used in this study were: $C_{11} = 84.4$ GPa, $C_{12} = 24.8$ GPa, $C_{112} = -80.5$ GPa, $C_{155} = -27.5$ GPa, and $C_{456} = -16.5$ GPa. From the relation between the third-order elastic constants, the following values can be deduced: $C_{111} = -190.4$ GPa, $C_{123} = -91.3$ GPa, and $C_{144} = 5.4$ GPa. The density of the glass was 2508 Kg m^{-3} .^{22,23} For the 3 sheets having undergone chemical tempering of 3, 16, and 24 h, named, respectively, CT3, CT16, and CT24, the superficial stresses were, respectively, -682 , -634 , and -535 MPa and the thicknesses of the superficially compressive fields of stress were, respectively, 13.4, 25.8, and $34.8 \mu\text{m}$. The theoretical dispersion curves were obtained over a range of frequencies up to 300 MHz and are reported in Fig. 2.

It is very interesting to analyze these curves as the influence of the level of superficial stress and its depth on the phase velocity of the surface wave can be observed. The phase velocity of the surface wave systematically decreases

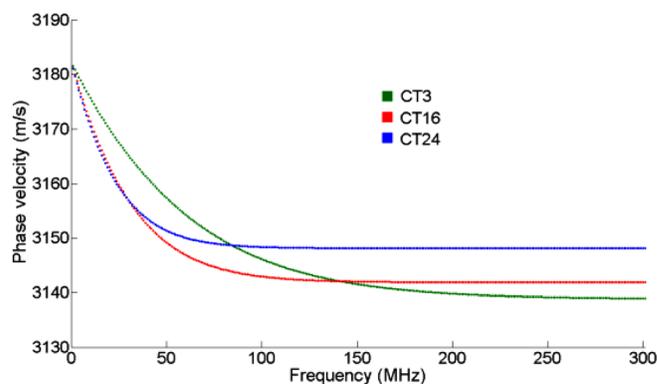


FIG. 2. Phase velocities of the first Rayleigh mode propagating on three chemically tempered glass sheets in the frequency range of 0-300 MHz.

in the presence of a superficial field of stress. The stressed cortical zone is called “loading” because it loads the substrate.²⁴ In this case, the velocity of the surface wave compared with the velocity of the surface wave which would propagate on an unstressed substrate is reduced by the presence of the superficial compressive stress. This phenomenon is consistent with the fact that the surface waves are slowed in the presence of a compressive stress.²⁵

Given that the longer the duration of immersion the greater the thickness of the stressed layer the more the stress decreases, it thus becomes possible to interpret the evolution of the first Rayleigh mode phase velocity as the frequency increases. Indeed, at low frequency, the wavelength of the surface wave is very big in comparison with the thickness of the stressed glass and the wave phase velocity essentially depends on the characteristics of the substrate. Second, it is the thickness of the stressed layer which will have the greatest influence on the wave velocity: the greatest decrease in velocity was observed with sheet CT24 immersed for 24 h (with the thickest stressed layer); this is revealed for the range of frequencies 0-25 MHz. At very high frequency (above approximately 150 MHz), the wavelength is in the order of the depths of the compressive stressed cortical zones, which means that the sheet of glass with the highest surface stress will slow the Rayleigh wave the most. The lowest phase velocities can be observed on the dispersion curves for sheet CT3, which has the highest surface stress.

SAW are thus dispersive on a stressed structure at micrometric scale and over a frequency range between 5 and 60 MHz and their phase velocities may decrease by a few tens of meters per second. So, if it is possible to carry out measurements over this wide frequency range, it is possible to accurately characterize the depth of the zone of compressive stress, which is why we have chosen to use SAW to highlight this phenomenon on these structures.

The main challenge was first to develop sensors to generate high frequency SAW over a wide frequency range and second to develop a signal processing method to obtain sufficiently accurate estimates of SAW propagation velocities. To meet these challenges, we proposed narrowband SAW-IDT MEMS, which offers the possibility of generating quasi-monochromatic surface waves covering a wide range of frequencies (5–60 MHz). It is thus possible to implement techniques for estimating phase velocities as with non-dispersive materials allowing rapid and accurate estimation of SAW

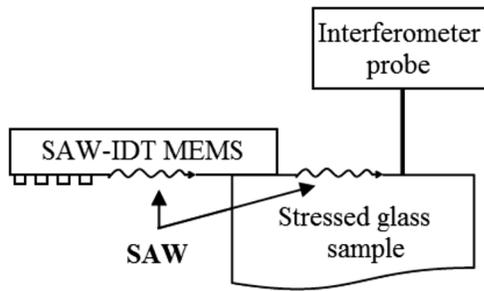


FIG. 3. Sketch of generation of surface acoustic waves with IDT sensor and detection with interferometer in stressed sample.

phase velocities.⁴ The IDT is placed flat and maintained on the sample with an acoustic couplant; the transducer surface, on which the fingers are deposited, is in contact with the sample. For SAW detection, a heterodyne interferometer was used. Good detection levels with good signal to noise ratios have been obtained thanks to the quality of the coupling (Fig. 3).

As an example, Fig. 4 presents the phase velocity measurements obtained for sheet CT24. For the 13 frequencies tested corresponding to the MEMS eigenfrequencies, the means of the phase velocities are represented by dots and the dispersion of these velocities (\pm standard deviation) by error bars.

An inverse method was developed in order to obtain the thickness of the superficially stressed layer for each of the 3 immersions. A least squares minimization was carried out in order to obtain theoretical propagation velocities as close as possible to the experimental values. The minimization method is based on the dichotomy. It consists of an iterative method of bracketing the desired minimum. Starting from an initial bounded interval, which is guaranteed to contain a minimum, the scanning of the interval with different small constant steps allows the width of the bounded interval to be progressively reduced until the desired convergence threshold is reached, i.e., until the width of the bounded interval is sufficiently small with a desired tolerance. To solve this inverse problem, 36 velocities were measured between 5 and 60 MHz. The theoretical values of the phase velocities were calculated from the stress values and initial thicknesses, the elastic constants and the density for each of the MEMS eigenfrequencies over the entire range of working frequencies. The processing algorithm was then sought to maximize the value of the coefficient of determination, R^2 .⁴ A large interval (1 μm to 101 μm) for the thickness was chosen to ensure an extremum of R^2 was obtained.

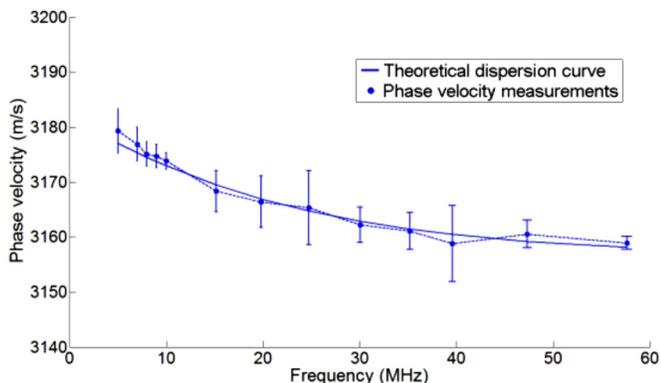


FIG. 4. Phase velocities on sheet CT24 and dispersion curve after inversion.

The results obtained for the 3 sheets CT3, CT16, and CT24 were 17, 25, and 34 μm , respectively, which are equivalent to deviations in μm of 3.6, 0.8, and 0.8, respectively, for the 3 immersions. The inversion results obtained relating to the depths of the stressed layers confirm the variation in the depth as a function of the duration of the chemical tempering. In addition, these values are in perfect agreement with those predicted by the tempering process implemented. The deviations in the thicknesses of superficially stressed areas are very low and the deviation observed with sheet CT3, although relatively low, could be improved by increasing the range of frequencies measured to over 60 MHz.

In conclusion, in this study we have shown that the behavior of a structure with superficial residual stress in relation to surface waves is analogous to that of a layer on substrate-type structure. The propagation of surface waves is thus addressed in the context of a classical formalism (second order) with effective elastic constants linked to the state of stress in the layer and the substrate. We have also shown that the depth of the stressed cortical zone can be estimated with good accuracy by resolving the inverse problem.

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