Non-destructive Characterization of Laminated Composite Films by Laser Ultrasonic Technique

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The method of using pulsed surface acoustic waves generated by pulsed laser to evaluate properties of layered films, such as Young’s modulus, density and thickness etc., has been investigated. A novel fcc/Ll₂ metal (Ni)-intermetallic (Ni 3Al) composite film is characterized, in which the Fourier transform of the pulsed surface acoustic wave signals yield the dispersion curve of the surface wave velocity spectrum. By theoretical simulation, the Young’s modulus about 284 GPa, and density about 7.650 g/cm³, as well as the thickness about 140 μm of the composite film have been obtained. For comparing, we also using this method to detect the single Ni₃Al film and obtain its Young’s modulus of 369 GPa. The Young’s module of the laminated composite film and the intermetallic (Ni₃Al) film are much larger than that of Ni. From these results we can infer that the laminated Ni-Ni₃Al film is expected to have better mechanical properties.

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The ultrasonic wave excited by a short laser pulse has been applied increasingly to investigate the mechanical and elastic properties, such as Young’s modulus, Poisson’s ratio and density of materials in recent years. For thin films or coatings, the mechanical and elastic properties are greatly affected by the film thickness and deposition technology. These properties may reflect the microscopic structure and quality of the films, which often deviate greatly from those of the bulk materials and difficult to be directly measured. By using laser ultrasonic technique to generate surface acoustic wave (SAW) on the film materials and then to detect the SAW signals, one can obtain the parameters quickly and conveniently¹, ².

When a pulsed laser illuminates on the surface of a sample, which prepared by a film deposited on a substrate, a SAW pulse is generated and propagates along the surface of the film. The parameters of the film may influence the propagation of the SAW. For higher frequency, the velocity of the surface wave may reflect the features in the near-surface region more sensitively than for those of the deep-surface region. Therefore a high enough frequency is needed for detecting thin films³. With wideband frequency SAW detection method, these parameters of the films can be simultaneously determined.

In this paper, the laser ultrasonic (SAW) technique is used to measure the mechanical and acoustic properties of a novel fcc/Ll₂ (a compound structure derived from fcc structure) metal-intermetallic composite film that consists of a composite layer deposited on a silicon (001) substrate. The composite film includes alternately deposited three sub-layers of nickel-aluminide (Ni₃Al) and two sub-layers of Ni⁴. The structures consisted of metal-intermetallics have been demonstrated to have great usefulness⁵, but the mechanical and elastic properties of this kind of layered composite films are still not very clear yet. In the experiments, the SAW pulses are detected by a piezoelectric transducer that pressed on a knife-edge⁶ and the detected signals are processed according to the algorithm that discussed later. For comparison, a single Ni₃Al film on the silicon substrate is also investigated, in which a larger Young’s modulus and better mechanical property of the film are obtained.

Samples

The laminated composite film we studied is an fcc/Ll₂ metal-intermetallic composite film. As we know, the intermetallics materials offer the advantages of higher specific strength and higher temperature capability, but lack ductility and fracture toughness⁷. By compositing these intermetallics with ductile metals, such as fcc metals, toughness may be improved, then the composite may have more applicability as supporting materials in micro-electromechanical systems and other industry fields. Besides, because of the good mechanical properties of Ll₂ intermetallics and small lattice mismatch between appropriate metals and intermetallics, the interface is expected to have favorable toughness and flexural strength.

The structure of the film studied is composed of a pure fcc element (Ni) and an ordered intermetallic compound (Ni₃Al), which are sputtered to the substrate alternately by the
magneto-sputtering technique. For Ni and Ni₃Al, their crystal lattice mismatch is 1.4%. Therefore, they can be well combined in the interfaces. When making the films, the sputtering energy of Ni is different with that of the Ni₃Al because of their distinct sputtering yields. The thickness of any single sub-layer is relative thick and the uniformity is difficult to be controlled during the sputtering procedure.

The SEM micrograph of the profile of the laminated composite film is shown in Fig.1. From the top there exist five alternate sub-layers of Ni and Ni₃Al totally, in which the thickness of the exterior layer is about 20μm. The bottom with some stripes is the substrate of silicon (001).

Experiments and Calculations

The experimental setup of the laser ultrasonic technique is shown in Fig.2. In the experiments, a Nd:YAG laser with nanosecond pulses (Continuum NP70) operating at 532 nm is used to excite the surface acoustic wave pulses at the surface of the sample. The laser beam is focused by a cylindrical lens and can be moved along the surface of the specimen, so the SAW pulses can be received in different distances. The frequency and amplitude of the SAW can be increased when using shortened and well-focused laser pulse. Be sure the energy of each laser pulse less than 1mJ to avoid the destruction of the surface of the sample when the focused beam width is less than 50μm. The laser-generated SAW signal is detected with a PVDF transducer. To obtain the required spatial resolution, the PVDF foil with the thickness of 9μm is pressed onto the surface of a stainless steel knife-edge. The contact area extends only several μm and is perpendicular to the propagation direction of the SAW. The distance between the transducer and the focused laser line can be varied from 5 mm to more than 1cm. The highest detection frequency of this system can be up to 100MHz, which depends on the material properties and the sizes of the laser beam and the transducer. For the relatively thick film, the highest frequency sometimes decreases to about 25MHz.

Experiments and Calculations

The theoretical fitting of the experimental result is to compare the calculated curves of different sets of parameters with the dispersion curve of experiment to search for a least square deviation. The set of parameters for the best fitting represent the solution of the inverse problem to determine the film properties. For a given frequency \( f \) and a set of given parameters, the phase velocity of the corresponding spectral component depends on a dimensionless factor \( kh \) that equals the product of the wave number \( k = 2\pi f/c(f) \) and the film thickness \( h \). Let \( f_{\text{max}} \) be the maximum frequency, the value \( k_{\text{max}}h \) may be an important judging standard of the computation. For larger values of \( k_{\text{max}}h \), two or more film parameters can be determined.
Results and Discussions

In the experiments of the Ni-Ni3Al laminated composite film, the distance between two detection points is 4 mm. To evaluate the film parameters, the signals are processed according to Eq.(1). The measured dispersion curve is shifted a little about less than 1% to fit the SAW velocity at the low frequency limitation that is well known as the SAW velocity of the substrate. Then, the parameters vary in a wide enough range to search for a set of rough estimated values of least square deviation. At last, applying these parameters as the initial values of Levenberg- Marquardt fitting method, which is a common used method of the nonlinear optimization, to find the accurate values. In order to avoid the local optimized solutions, the estimated value of the thickness according to atomic weight of the elements are used as the initial values.

In Fig.3 the measured dispersion curve and the calculated best fits curve for single-layered Ni3Al film are shown. The fitting results yield the density of 7650 kg/m², Young’s modulus of 284 GPa and Poisson’s ratio of 0.26. The thickness is close to the value observed by SEM that is about 150μm. Coincident with the relatively large thickness of the composite film, the central frequency of the signals is about 10 MHz. The maximum value of \( k_{\text{max}} \) reaches to 2.8 and meets with the multi-parameters fitting condition. For the film has strong attenuation, the upper frequency limitation is much lower than the bandwidth of the experimental system.

In order to compare the performance of the composite film with those of the sub-layers, it is important to know the parameters of Ni and Ni3Al. Since there is no published Young’s modulus and Poisson’s ratio of Ni3Al materials, we detect the SAW propagation of Ni3Al film on Si substrate that is made by the same sputtering technology. Also the analysis procedure for the laminated film is applied to the single-layered Ni3Al film. The measured and calculated dispersion curves are shown in Fig.4. The parameters of Ni3Al film are yielded, in which the density is 7380 kg/m³, thickness is 39μm, Young’s modulus is 369 GPa and Poisson’s ratio is 0.18. The highest frequency of the wave propagating along the surface of the single Ni3Al film is 25 MHz and the maximum value of \( k_{\text{max}} \) is about 1.6. For comparison the parameters of both the composite and the single films are listed in Table 1.

From Table 1, it is evidently that by compositing these highly strengthen intermetallic Ni3Al with ductile metals Ni, the flexural strength and toughness of the film can be improved. The laminated structure of multi-layered composite film may expect to simultaneously possess the advantages of fcc metals and Ll2 intermetallic, i.e. good flexural strength and toughness. According to the measured results, the Young’s modulus of the laminated composite film is between those of Ni and Ni3Al. The mechanical property of this film is greatly improved comparing with metal nickel. Therefore the fcc-Ll2 metal-intermetallics laminated composite film may have better comprehensive mechanical behavior than metal films and silicon-based single layered films. The metal-intermetallics composite films can be developed as new materials in applications of miniaturized sensors and micro-machines because of its enhanced mechanical capability.

As the conclusion, combined the SAW spectroscopy with the calculation of the dispersion curve of the SAW velocity, as well as with the least square fitting method, the laser ultrasonic technique can be an effective and convenient way to obtain the mechanical parameters, such as Young’s modulus and Poisson’s ratio of thin film materials. We can also employ the laser ultrasonic technique to design and monitor the mechanical properties of the structure of the films, specially for laminated composite films.

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References