Mechanical Characterization of Coatings at Elevated Temperatures Using Laser-Induced Surface Acoustic Wave Spectroscopy

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PSE 2022

Laser-induced surface acoustic wave spectroscopy allows quick and non-destructive characterization of coatings and surfaces since acoustic properties are strongly correlated to elastic properties, coating thickness, but also crack density. Therefore, the method is a quick and powerful tool for surface characterization and can be found today in research and development, quality control and as a precise and scientific reference method. However, measurement close to room temperature limits its potential to study high-temperature effects as found in applications.

Surface Acoustic Wave Spectroscopy

Surface acoustic wave (SAW) spectroscopy makes use of different propagation speeds in the surface-near region (e.g. coating) and the bulk (e.g. substrate). SAW penetration depth depends on their frequency. Low frequencies will propagate deep in the material, mainly influenced by the substrate. High frequencies will propagate close to the surface, mainly influenced by the coating.

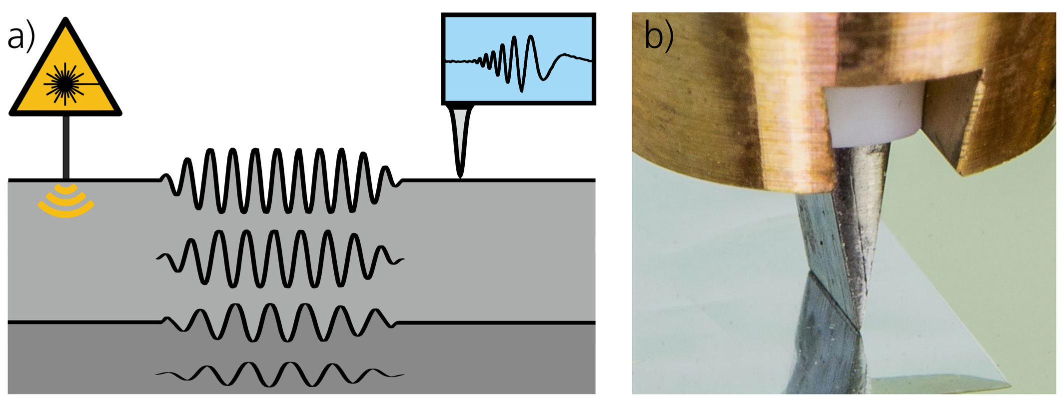

Figure 1: a) Scheme of surface acoustic waves generation by a short laser pulse on the surface (left), propagation through coating and substrate (center) and detection by a piezo-electric contact sensor (right), b) close-up image of contact sensor on piezo-electric foil and silicon wafer

Generation of SAWs is accomplished contact-less with a short laser pulse on the surface of interest, while measurement of the SAWs is performed with a piezo-electric mechanical contact sensor (Figure 1a), where the sensor is gently pushed against a piezo-electric polymer foil laying on the substrate.
Even though contact-less measurement options exist, e.g. based on laser interferometry, piezo-mechanical contact sensors show significant advantages in signal intensity, roughness tolerance and cost. Such high frequency contact sensors, consisting of a low-radius steel wedge pushed on a polymer-based piezo foil (Figure 1b), however, have a limited temperature range of about 80°C so that many coating scenarios with higher application temperature, e.g. tribological coatings for cutting or combustion engines, cannot be measured under such condition.

High temperature setup

Based on a standard LAwave® measurement system for SAW spectroscopy, a modified setup for higher temperatures was developed. While in the former setup the sample was moved with respect to the fixed laser, using a high precision translation stage, now the laser including all optics was moved with respect to the fixed sample. That allowed incorporation of an electric heating table, where temperature could be kept within 1 K of the desired value up to 600°C maximum temperature.
Wedge-shaped sensors were manufactured with conventional geometry from 16MnCr5 steel and subsequently coated with piezo-electric aluminum nitride (AlN) coatings, using magnetron sputtering. Coating parameters were adjusted to maintain good adhesion on the strongly curved wedge as well as highly orientated crystal growth to achieve high piezo-electricity.

Evaluation of setup

AlN coated sensors were studied in cross section to verify coating thickness and good adhesion (Figure 2). Subsequently, the sensors were tested in the modified setup using a single crystal polished silicon wafer as reference sample. Advantages of such material are high temperature stability, low attenuation over a wide frequency band and absence of any dispersion. In that best case scenario signal properties are limited by the measurement system rather than the sample.

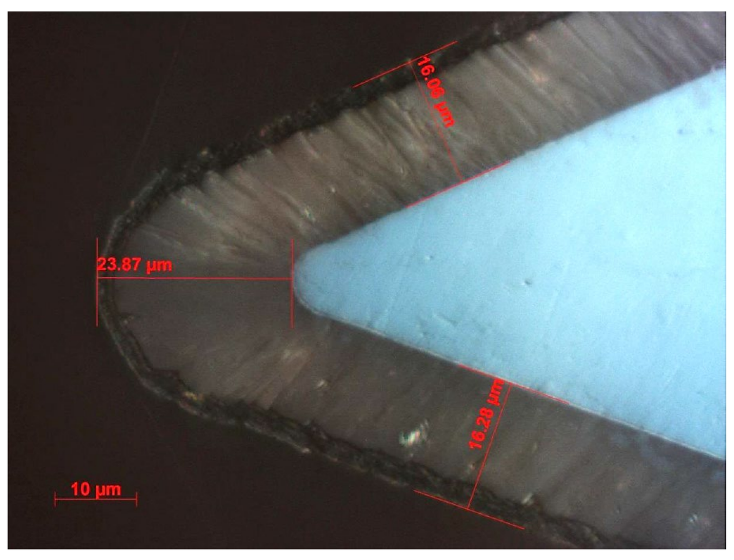

Figure 2: Cross section of AlN coated sensor wedge, showing a tilted columnar crystal structure and a coating thickness of approx. 24 µm at the line of contact

Signal properties are evaluated by maximum frequency of the pulse and maximum peak-to-peak amplitude of the piezo-electric signal, where higher values are most favorable.

The AlN coated sensor was compared to two conventional sensor wedges with piezo foil. As the manufacturing geometry of the edge has a strong influence on the performance, average achievable and best achieved preparation conditions were used as references. Results of evaluation are shown in Figure 3. At room temperature, the peak-to-peak voltage was below 0.5 V and thus well below voltages achievable with a piezo foil. However, the signal intensity was still sufficient for further evaluation and could be maintained over the full temperature range up to 600°C.

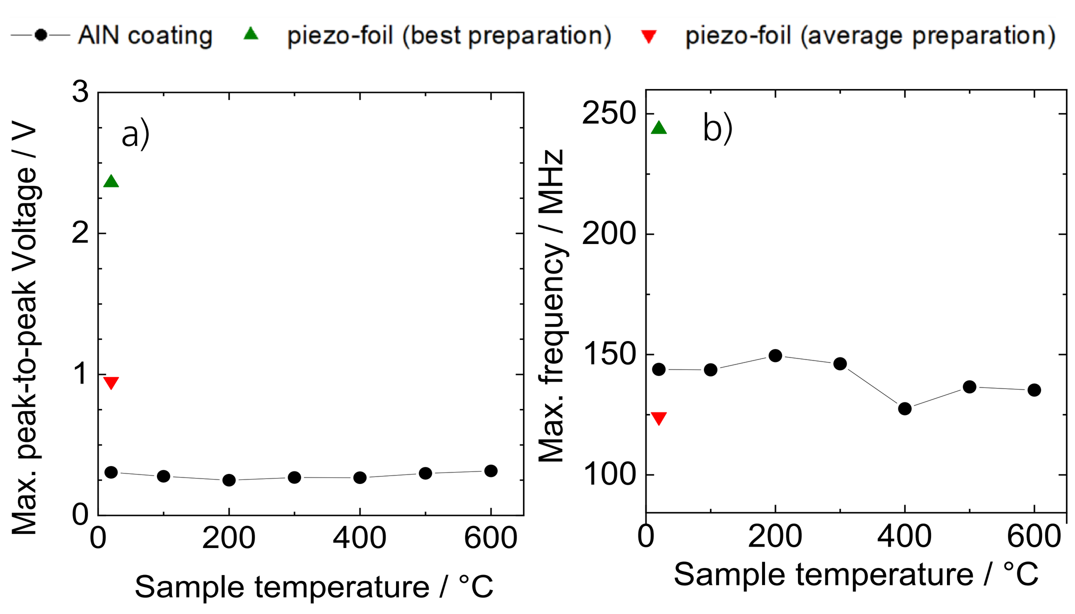

Figure 3: Comparison of AlN coated sensor performance up to 600°C with conventional setup using a piezo foil and an average and best prepared sensor geometry at room temperature. Maximum peak-to-peak voltage of the piezoelectric signal (a) and maximum measurable frequency (b) is shown.

Maximum frequency of the AlN coated sensor was well below best preparation, but somewhat better than average preparation. Maximum achievable frequency was stable up to 600°C and in the range of 125-150 MHz, which is sufficient for coating in the range of 1 µm and thicker, which includes common PVD coating applications.

Application examples

Temperature resistance of ta-C coatings
Superhard tetrahedral amorphous carbon (ta-C) coatings are often used as tribological coatings, also at elevated temperature. So far, their mechanical temperature stability has not been studied in detail. Therefore, a-C (25% sp^3) and ta-C (50% and 75% sp^3) coatings were tested at increasing temperature until the signal was lost, corresponding to a loss in mechanical integrity. Results shown in Figure 4 find that softer a-C coatings are stable up to 350°C, while harder ta-C coatings keep high modulus and full mechanical integrity up to 550°C in air atmosphere.

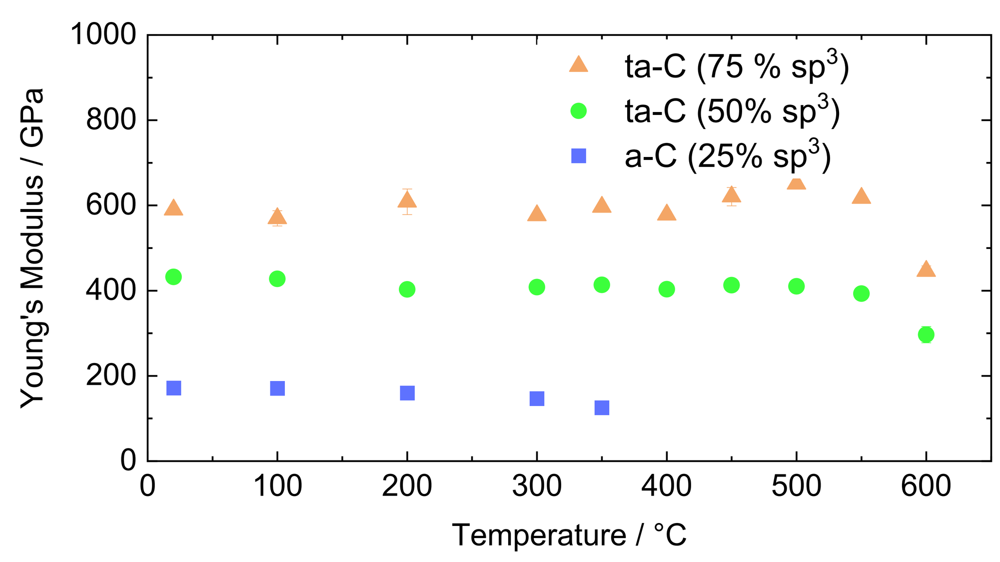

Figure 4: Young's Modulus of a-C and ta-C coatings with different sp^3 ratios, measured at temperatures up to 600°C.

High precision Young's Modulus measurement
Using nanoindentation, Young's Modulus of coating is measured perpendicular to the surface after plastic deformation, leading to compressed defects, pores and cracks. In contrast, SAW spectroscopy allows measurement of the effective „engineering" modulus parallel to the surface in the purely elastic regime, including all native defects (Figure 5).

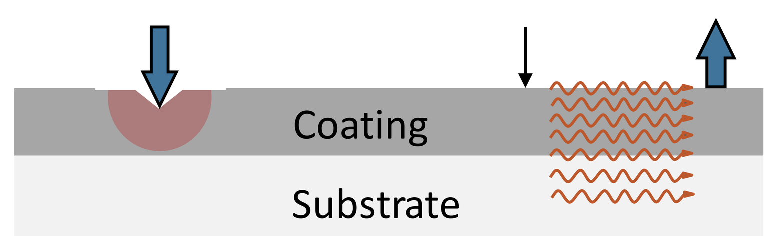

Figure 5: Principle of measurement for nanoindentation (left) and SAW spectroscopy (right)

Especially in coatings with columnar or porous structure, in-plane modulus can be significantly lower, leading to earlier failure than expected, when using FEM or analytical stress calculation tools to understand thermal and intrinsic stresses or crack initiation.

Conclusion and Outlook

In this work it was shown that the advantages of surface acoustic wave spectroscopy for characterization of coatings and bulk materials can be extended to at least 600°C, using a modified measurement setup and newly developed AlN coated contact sensors. It now is possible to study the effect of thermal activated processes like oxidation, diffusion and phase transitions on mechanical properties. Furthermore, it is possible to measure Young's modulus with highest precision not accessible with other methods.
As AlN maintains piezoelectricity to temperatures above 1000°C, it might be possible to extend the temperature range well beyond 600°C with further adjustments on the measurement system.
Overall, SAW spectroscopy of coatings and surfaces at elevated temperatures opens up many new possibilities for research and development.