Bulgarian Society for NDT
International Journal "NDT Days"
Volume VI, Issue 2, Year 2023

ISSN: 2603-4018
eISSN: 2603-4646

Curing Monitoring in Metal-Polymer Structures by Laser-Induced Ultrasound

Vitaliy K. MERINOV[1], Victor V. KOZHUSHKO[1], Vladimir P. SERGIENKO[1], Alexander R. ALEXIEV[2]

[1] V.A. Belyi Metal-Polymer Research Institute of National Academy of Sciences of Belarus, Gomel, Belarus
e-mail: vkozhushko@mail.ru

[2] Institute of Mechanics at the Bulgarian Academy of Sciences, Sofia, Bulgaria,
e-mail: alexiev@imbm.bas.bg

Abstract
The possibilities of short ultrasonic pulses for measuring the transmission of layered metal-polymer structures and for monitoring the curing process in a polymer layer were demonstrated. Probing ultrasonic pulses were excited by means of optoacoustic conversion also known as laser ultrasound. The nanosecond laser operating in the Q-switched mode at a wavelength of 532 nm was employed. The absorption of optical radiation in a thin near-surface layer creates thermoelastic stresses, as a result of which a longitudinal pulse was excited with a pronounced compression phase and a broadband spectrum covering the frequencies range from 0.5 MHz to 50 MHz. Ultrasonic pulses passed through the layered structure and were detected by a broadband piezoelectric transducer from the opposite side of sandwich structure. The sensitive element of the transducer was made of a polarized film of polyvinyldylene fluoride (PVDF) with a thickness of 25 μm and a diameter of 3 mm.

Keywords: Laser-induced ultrasound, time of flight, epoxy resin, aerospace, layered sandwich structure

1. Introduction

Metal-polymer layered structures allow reduce weight and vibration of construction that is crucial for aerospace applications. The curing time is an important parameter of sandwich technology. During the hardening, the physical and mechanical properties of the polymer component are changing, in particular, the rigidity, which affects the speed of ultrasound and therefore the time of propagation of elastic pulses through the layer of adhesive. Measurement of the characteristics of ultrasound transmission by such a structure can be used to solve the tasks of quality control of adhesive joints and monitoring the process of resin polymerization. This paper suggests the solution by means of laser-induced ultrasonic pulses. The excitation of short-time probe ultrasound is carried out by ten nanosecond laser pulses illuminating the metal surface of the sandwich structure. The detection of the transmitted ultrasound has been done by a piezoelectric PVDF film transducer. This approach allowed non-contact excitation of broadband ultrasonic pulses and analysis with high temporal resolution, while the pressure pulses can be measured by cost effective traditional contact or immersion methods in comparison with all-optical technique [1-3].

2. Experiment

The metal-polymer structure comprised of two D16 (Russian notation) aluminum plates and a adhesive layer of two-component epoxy glue Scotch (3M, Taiwan). The thickness of the aluminum layers is 2 mm, the epoxy layer has been placed between aluminum plates separated by a steel washer with an internal diameter of 8 mm and a thickness of 1.3 mm that fixed the distance between the plates. The curing of epoxy occurred at room temperature of 22 ºC during the experiment. The measurement of laser-induced ultrasonic pulses is carried out by the immersion method according to the shadow scheme shown in Fig. 1.

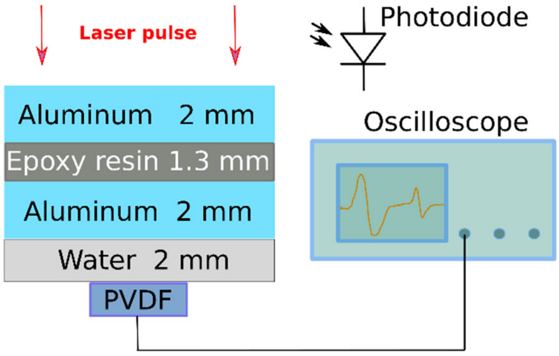

Fig. 1. Scheme of experimental arrangement

The radiation of an LS-2131M-10 laser (Lotis TII, Belarus) operating in the Q-switched mode illuminated the upper aluminum plate. The energy of a single laser pulse was 20 mJ, and the duration was < 12 ns. The power density of laser radiation did not exceed 10 MW/cm2, which corresponded to the thermoelastic regime of ultrasound excitation [2]. Absorption of the laser radiation with a wavelength of 532 nm in the metal occurred over the area of the light spot at a depth of no more than a few tens of nanometers that led to local heating of the near-surface volume. During the laser pulse heat penetrated into the bulk of aluminum to a depth of approximately 1 µm, the estimated temperature raise is above 100 ºC. Fast heating induced expansion and thermoelastic stresses, the relaxation of which caused ultrasonic pulse. Without radiation focusing in the thermoelastic mode of optoacoustic conversion, longitudinal waves are excited most efficiently compared to shear, surface and guide waves. In the experiments, the diameter of the laser spot was 5 mm, the diameter of the sensitive element of the piezoelectric transducer was 3 mm that allowed to neglect the influence of diffraction during the propagation through the sandwich structure. The exited ultrasonic pulse possesses pronounced compression phase of about several tens of nanoseconds duration that provides high temporal resolution for velocity measurement tasks. An excited probing pressure pulse propagated to the rear side of the layered structure. A layer of 2 mm thick distilled water between the structure and the piezoelectric transducer provided acoustic contact. The transducer operates only as receiver of ultrasound. The transducer is based on 25 µm thick polarized polyvinylidene fluoride (PVDF) film (Piezotech, USA). The construction of transducer allowed broadband detection of ultrasonic pulse. The preamplifier was built into the transducer housing operated in short-circuit mode that ensured registration of the frequencies with an upper limit reaching 90 MHz [4].

The pulses were recorded using a DS1104Z digital storage oscilloscope (Rigol, China), with its two channels involved, the digitization frequency was 500 MHz. The oscilloscope was triggered by the rising edge of the electrical signal of PIN photodiode S5971 (Hamamatsu, Japan), which determined the moment of irradiation of the metal surface and provided the beginning of time interval of ultrasound propagation through the sandwich structure and water layer to the PVDF receiver. An example of the signal measured 30 min. after start of experiment is presented in Fig. 2.

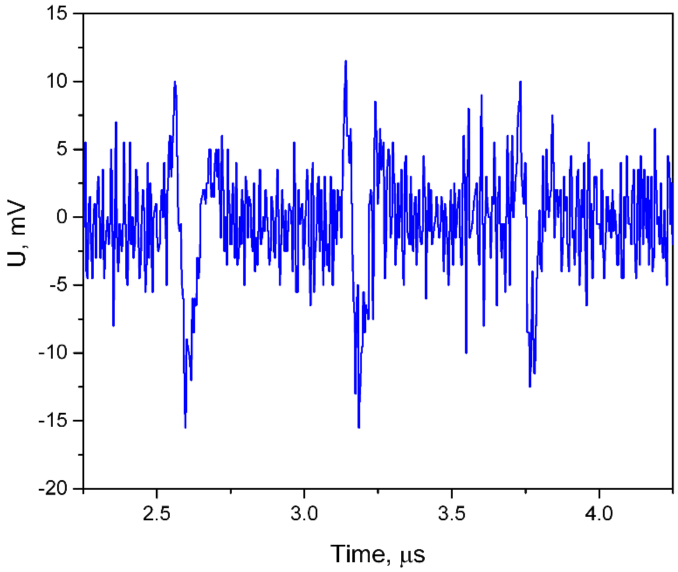

Fig. 2. Ultrasonic signal measured 30 min. after curing start

In the course of the experiment, for a sequence of measured optoacoustic signals, the energy of laser pulses was fixed at a level of 20 mJ, which provided conditions for stable excitation of ultrasound in aluminum. The curing of the epoxy causes an increase of its elastic moduli and acoustic impedance of the material. The determination of the degree of change in the mechanical characteristics of the resin during polymerization was carried out by the propagation time of ultrasonic pulses through the sandwich structure. The measurements of the transmitted ultrasonic signals were carried out with a variable time interval.

As it is shown in Fig. 2 the profile of detected pressure pulse yielded a bipolar form, starting with a positive and ending with a negative peak with a characteristic descending edge between them. The travel time of ultrasound through the layered structure was determined from the midpoint of the descending front of the pressure pulse. The dependencies obtained for the aluminum / epoxy resin / aluminum structure are collected in Fig. 3, where for the clarity only parts of the signals overlapping the first falling fronts are shown.

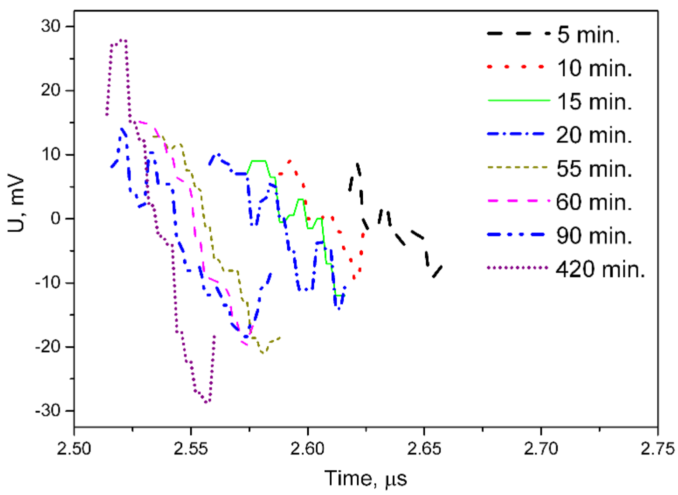

Fig. 3. The sequence of the first falling frons of ultrasonic pulses during epoxy resin curing

The time of arrival of the first registered pulse in a series of measurements was used. The onset of measurements was 5 minutes after the production of the sandwich structure, the next time is on the right in Fig. 3. The falling front of the first measurement is located before 2.65 μs, the peak-to-peak amplitude of the pulse is a bit less than 20 mV. The arrival time shifts to the left and the peak-to-peak amplitude exceeds 20 mV after 30 min. An hour later, there is the further increase of the peak-to-peak amplitude up to 35 mV. The signal recorded after 7 hours has the peak-to-peak amplitude of approximately 60 mV. So the falling fronts of pressure pulses gradually shift to the left side of time axis with an increase of peak-to-peak amplitude, that is explained by a changes of the mechanical properties of the epoxy resin.

The reference value of the cured epoxy ultrasound speed is 2.5 km/s. Based on the results of the measured sequence, a decrease in the arrival time of the falling front of the first pulse by 100 ns was established. As the thickness of epoxy layer is known the estimated ultrasound velocity in the liquid uncured resin is approximately equal to 2.0 km/s. Polymerization increases the speed due to the hardening of resin and growing stiffness. The velocity also affects the acoustic impedance, which is a product of density and speed of ultrasound in the material. Reference value of aluminum acoustic impedance is approximately 17×10^6 Pa s/m, impedance of cured epoxy resin and water are 2.95×10^6 Pa s/m and 1.49×10^6 Pa s/m, respectively [5]. The increasing of the resin impedance reduced the transition loss across the aluminum / epoxy resin / aluminum interfaces.

3. Conclusions

Thus, it is shown that the measurement of the amplitude dependence of laser-induced ultrasonic pulses makes it possible to monitor the curing process of the inner epoxy resin layer of sandwich structures via the decrease of arriving time which is defined by elastic properties of polymer. The suggested experimental arrangement can be applied to optimize the technology of metal-polymer sandwich structures and to developed nondestructive method to assess the mechanical properties of the adhesive layers and the quality of adhesive joints.

Acknowledgments
This research was supported by program “BRFFR-MIDRU-2022”, grant T22UZB-069.

References

1. Zhang Y. et al. Research on epoxy resin curing monitoring using laser ultrasonic. Measurement, Vol. 158, 107737, 2020, doi: 10.1016/j.measurement.2020.107737
2. Meng J. et al. Laser ultrasonic inspection for mechanical properties of materials at high temperature. J. Appl. Phys., Vol. 134, 053101, 2023, doi: 10.1063/5.0147615
3. Haderer W. et al. Spatial imaging of stratified heterogeneous microstructures: determination of the hardness penetration depth in thermally treated steel parts by laser ultrasound. NDT & E Inter., Vol. 138, 2023, doi: 10.1016/j.ndteint.2023.102868
4. Kozhushko V.V., Paltauf G., Krenn H. Hysteresis-like attenuation of ultrasound in severely plastically deformed nickel. J. Phys. Conf. Ser., Vol. 278, No. 1, 2011. pp. 012012-1-4. doi: 10.1088/1742-6596/278/1/012012
5. Fu Y., Kabir I. I., Yeoh G. H., Peng Z. A review on polymer-based materials for underwater sound absorption. Polymer Testing, Vol. 96, 2021, doi: 10.1016/j.polymertesting.2021.107115