Nonlinear ultrasonic detection for curing properties of the liner

JIANG Nian[1], WANG Zhao-ba[2]

(1. *Huazhong Institute of Electro-Optics, Wuhan National Laboratory for Optoelectronics, Wuhan 430223, China*;
2. *School of Information and Communication Engineering, North University of China, Taiyuan 030051, China*)

Abstract: The relationship between ultrasonic nonlinearity and microstructure of the liner was studied during the whole curing process by ultrasonic transmission method and infrared spectroscopy. Nonlinearity of input instrumentation was minimized by the natural filtering effect of piezoelectric discs and the maximum excitation energy was acquired simultaneously so as to improve the accuracy of the measuring data. The experimental results indicate that in the liner curing reaction at 40 ℃ ultrasonic nonlinearity parameter decreases gradually after a sharp decline, which is consistent with the outcome of infrared spectroscopy as the curing degree increases. The research suggests an effective nondestructive approach to detect the curing properties of the liner in a nonlinear ultrasonic way.

Key words: liner; curing properties; nonlinear ultrasound; nondestructive detection

CLD number: V435; O221.3 **Document code**: A

Article ID: 1674-8042(2018)04-0321-05 **doi**: 10.3969/j.issn.1674-8042.2018.04.003

0 Introduction

In the solid rocket motor, the liner between the shell and the propellant acts as heat insulating structure to protect the shell from direct erosion and ablation coming from high temperature propellant gas[1]. The adhesive quality of shell/liner directly affects the safety and performance of the engine. It is easier for traditional ultrasonic nondestructive testing to detect the complete debond flaw on shell/liner/propellant interface, but it becomes difficult to detect defects such as poor adhesive interface and kissing bond[2-4]. Recent studies show that the nonlinear effect is closely related to the curing state of the liner and the change of its mechanical properties[5-8]. Wu, et al. proved ultrasonic nonlinear coefficient could be used as a bridge between macroscopic and microscopic characterization of mechanical properties of materials degradation[4]. Hirsekorn S pointed out that under the finite amplitude ultrasonic wave excitation, the nonlinear stress-strain relation of the liner medium would lead to distortion of ultrasonic wave form and creating of high harmonic signal[9]. Therefore, higher harmonics in a monochromatic longitudinal wave give information about the curing state of the liner.

This paper presented a method of monitoring the nonlinear ultrasonic coefficient of the liner between the shell and the propellant during the curing process using transmitted p-wave. By comparing the results of ultrasonic monitoring and infrared spectroscopy, the nondestructive detection of the curing state of liner is achieved.

1 Theory of nonlinear ultrasound

Under the monochromatic finite amplitude longitudinal ultrasound excitation, nonlinear equation in isotropic materials is

$$\rho\frac{\partial^2 u}{\partial t^2}=\left(K_3-\beta K_2\frac{\partial u}{\partial x}\right)\frac{\partial^2 u}{\partial x^2}+\delta\frac{\partial}{\partial t}\left(\frac{\partial^2 u}{\partial x^2}\right)+F_{\mathrm{ext}}(x,t), \tag{1}$$

where ρ represents density, $u(x,t)$ is the particle displacement on x axis, $K_2=\rho c^2$ means the second order elasticity, K_3 is the third order elasticity, $\beta=-(3K_2+k_3)/K_2$ represents the nonlinear coefficient of material, δ is sound attenuation factor and $F_{\mathrm{ext}}(x,t)$ is external force per unit area.

For semi-infinite material ($x>0$), when the external force exerted on the boundary is the single-

Received date: 2018-07-17

Foundation items: National Natural Science Foundation of China (No. 61201412)

Corresponding author: JIANG Nian (jiang_nian@yeah.net)

frequency sine wave, according to perturbation theory[10], the approximate solutions of nonlinear Eq. (1) can be got by

$$u(x,t)=\underbrace{\frac{1}{8}k_0^2A_1^2x}_{A_0}+A_1\sin(-k_0(x-ct))-\underbrace{\frac{1}{8}\beta k_0^2A_1^2x}_{A_2}\sin(-2k_0(x-ct))+\cdots, \quad (2)$$

where wave number $k_0=2\pi f_0/c$, f_0 is the frequency of the sinusoidal signal, x is the transmission distance, A_0 represents DC rate, A_1 represents the fundamental frequency of the signal amplitude, and $A_n(n>1)$ is the nth harmonic.

Therefore, the nonlinear coefficient of the material can be calculated by

$$\beta=\frac{8}{k_0^2x}\frac{A_2}{A_1^2}. \quad (3)$$

The relative nonlinear coefficient of the material is

$$\beta'=\frac{A_2}{A_1^2}. \quad (4)$$

Comparing Eqs. (3) and (4), for the exact frequency f_0 and transmission distance x, relative nonlinear coefficient β' is proportional to β, so β' is often used to replace β.

2 Specimen and ultrasonic detection method

The specimen to be tested was made of 80 mm× 65 mm steel sheet/insulation adhesive structure, the first layer was a 4 mm steel sheet, the second layer was a 0.5 mm liner and the third layer was a 2 mm insulation layer. The liner was compounded of hydroxy-terminated polybutadiene (HTPB), toluene di-isocyanate (TDI) and accessory ingredient according to a certain proportion, and it should be stirred by heating magnetic stirrer to make sure exclude bubbles before use.

The p-wave transmission method was used in the experiment, and the proposed detecting system is shown in Fig. 1. The system includes function generator (RAM-SNAP), matched impedance, high-energy low-pass filter, attenuator, transducer, oscilloscope, computer, online test specimen and fixture. Temperature control system was primarily used to control the liner curing temperature and to refrigerate the transducers.

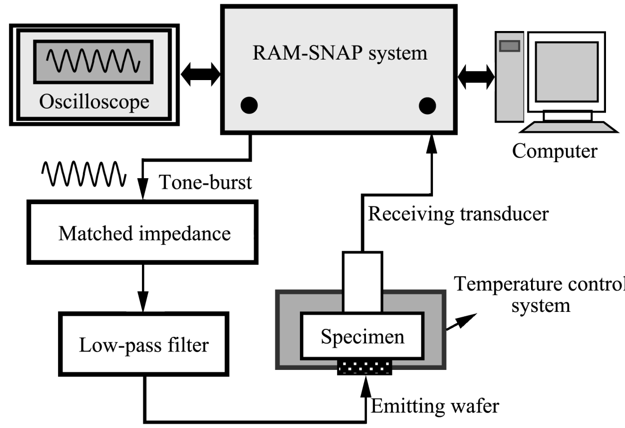

Fig. 1 Experimental setup of nonlinear ultrasonic detection system

The amplitudes of the fundamental and the second harmonic wave were measured by transmission method in this experimental system. A tone burst signal at 5 MHz from a function generator was fed into the matched impedance during the liner curing. Then the signal passed through a high energy low-pass filter to filter high frequency interference signal created by radio frequency (RF) gate. Piezoelectric disc, with center frequency of 5 MHz, was used as a transmitter and commercial broad-band; with center frequency of 10 MHz, it was used as a receiver. Two transducers were longitudinal wave right angle probe, and the effective diameter was 10 mm.

The transducers were coupled to the specimen with salol. The fixture was used to keep two transducers on the same centerline axis. The transmitted ultrasonic wave was averaged 256 times with oscilloscope, and then transferred to a computer for further signal processing. The fundamental and the second harmonic signal amplitudes were measured by fast fourier transform (FFT).

In order to reduce the influence of the nonlinearity from the function generator, low-pass filter and coupling agent, the transmitting transducer could be used as a natural filter[11]. Fig. 2 shows the frequency response of the piezoelectric disc used as the transmitter. In nonlinear measuring, the frequency close to a resonant peak in the frequency response was usually selected to maximize the excitation level within the specimen. Fig. 3 shows the nonlinear coefficient due to the transducer characteristics at the harmonics compared to that at the drive frequency. It can be seen from Fig. 3 that for the transducer frequencies of 2.3 MHz and 5 MHz, both result in minimizing the influence of apparatus-induced nonlinearity and at the same time maximizing excitation energy.

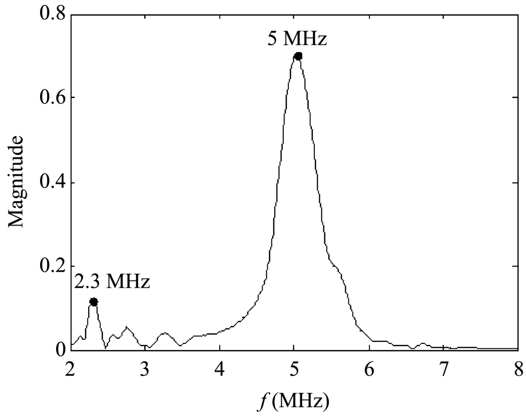

Fig. 2 Frequency response of piezoelectric disc

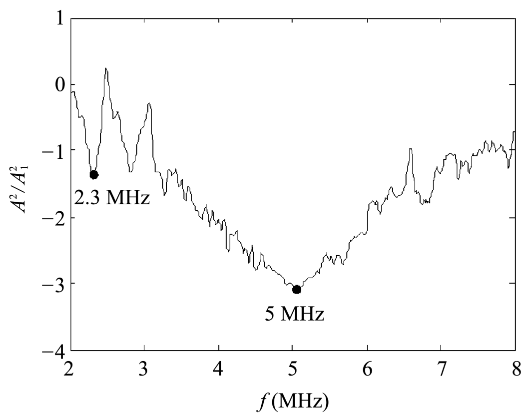

Fig. 3 Filtering effect of transmitting transducer

3 Infrared spectroscopy experiment

Liner sample was equably smeared on KBr salt tablets, making sure the coat was transparent. The salt tablets was then put into temperator at 40 ℃ and measured once every ten minutes by Thermo Nicolet Corporation Fourier transform infrared spectrometer (FTIR, Impact420). Fig. 4 shows the FTIR spectra of the liner at different curing times at 40 ℃.

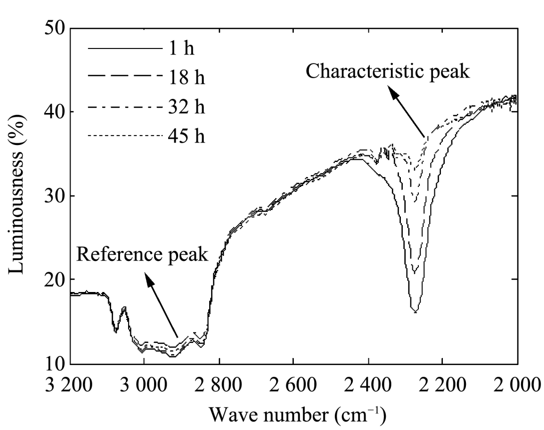

Fig. 4 FTIR spectra of the liner at different curing times

During the curing reaction, the concentration of —NCO grope decreases as —NCO group gradually reacts with —OH. On the contrary, the concentration of $—CH_3$ remains the same because it does not participate in reaction. As a result, —NCO (characteristic peak) and $—CH_3$ (reference peak) are suitable for quantitative analysis. Assuming that the transmittance of the characteristic peak is s_0 and the transmittance of the reference peak is $s_0^{'}$, the ratio of them is C, namely

$$C = \frac{s_0^{'}}{s_0}. \tag{5}$$

According to the Lambert-beer law, absorption peak intensity is proportional to its concentration. Therefore, parameter C can reflect the change of —NCO concentration in TDI revealing curing depth of the liner and its bonding properties.

4 Result and analysis

The bonded shell/liner/propellant specimen was monitored online using nonlinear ultrasonic measurement system.

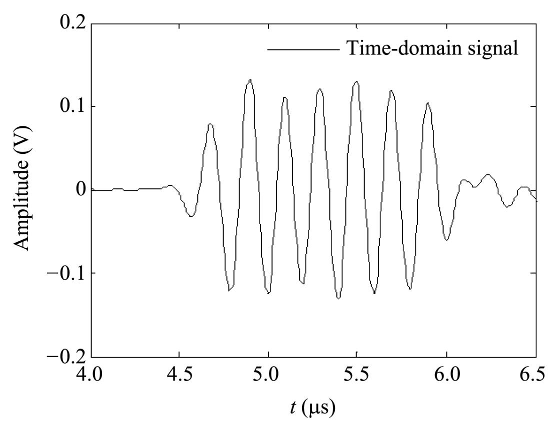

Fig. 5 Example of transmitted time-domain signal

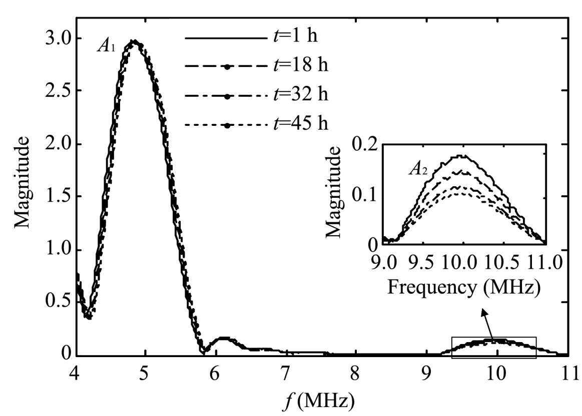

Fig. 6 Fundamental and 2nd harmonic amplitudes at different curing times

Fig. 5 shows an example of a transmitted time-domain signal which has become distorted. Fig. 6 shows the Fourier spectra of transmitted signal at different curing reaction times, including fundamental amplitude and the second harmonic amplitude. Compared with the fundamental amplitude, the second harmonic amplitude is very small. Fig. 6 shows the second harmonic clearly in the fundamental wave figure.

In order to visually reveal the curing properties of the liner, curing strength was tested while monitored by nonlinear ultrasonic approach. The curing properties of the liner at different curing times are shown in Table 1.

Table 1 Curing properties of the liner at different curing times

Curing time (h)	State of liner	σ (MPa)
4	Sticky, wiredrawing	0.83
6	Slightly sticky	0.95
10	Softer, inelastic	1.12
16	Clastic	1.08
25	Elastic	1.14
40	Elastic	1.16

Fig. 7 shows the results of normalized nonlinear coefficient (NLP) and infrared spectroscopy at curing temperature 40 ℃.

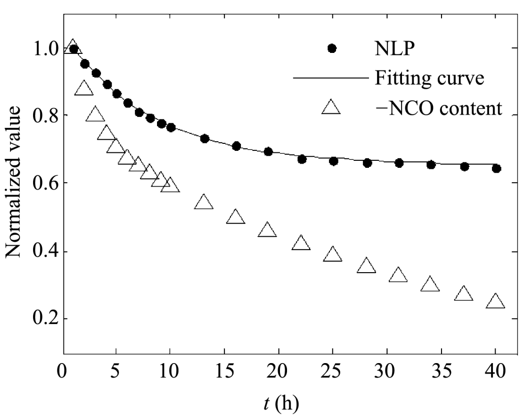

Fig. 7 Normalized relative nonlinear parameter and the content of —NCO vs. curing time

The —NCO group that reacts with —OH causes the concentration to decline rapidly and viscosity to increase during the first 10 h reaction. Network structure has already formed in the liner and the modulus of the liner basically remains the same after 10 h. Although the concentration of —NCO group (about 30%—40%) continues to decline, the bonding strength of the liner changes a little according to the data in Table 1. The chemical crosslink density of the interface between the liner, the shell, and the propellant increases rapidly during the first 10 h reaction. At the same time, the influence of nonlinear stress-strain relation on ultrasound decreases significantly, and the NLP falls off rapidly with increasing curing time. On the whole, chemical crosslink come to the end for the next few hours, the mild descent of NLP reveales that curing properties of liner changes a little, this phenomenon is in accord with the law summarized by Ref. [12].

Infrared spectroscopy technology reflects the curing process of the liner through chemical reactions. For the curing process of the liner includes not only chemical reactions but also physical crosslink, by measuring the nonlinear stress-strain interaction of the ultrasound with the microstructure of the liner, we can reasonably reveal the changes of bonding properties during its curing process.

5 Conclusions

1) In this work, the curing process of solid rocket motor liner is monitored using nonlinear characteristics of transmitted ultrasound wave. The NLP rapidly decreases, and then slowly declines during the curing reaction at 40 ℃. The change law of the liner is in accord with the result of infrared spectroscopy detection, which indicates that nonlinear ultrasonic approach can truly characterize the changes of bonding properties during its curing process.

2) The frequency response and filtering effect of piezoelectric disc show that transmitting transducer can maximize the excitation level at the resonant frequency of 5 MHz, minimize the input instrumentation nonlinearities and improve measurement accuracy simultaneously.

References

[1] Fan S, Wang Z B, Jin Y, et al. Design of automatic ultrasonic detection system for insulating layer debonding flaws of SRM. Journal of Astronautics, 2010, 31(6): 1646-1650.

[2] Vijaya K, Murthy C L. Some studies on evaluation of degradation in composite adhesive joints using ultrasonic techniques. Ultrasonics, 2013, 53(6): 1150-1162.

[3] Shui G S, Wang Y S. A method nonlinear ultrasonic evaluation of coating damage on metallic material. Journal of Solid Rocket Technology, 2012, 35(5): 703-706.

[4] Wu B, Yan B S, He C F. Nonlinear ultrasonic characterizing online fatigue damage and in situ microscopic observation. Transactions of Nonferrous Metals Society of China,

2011, 21(12): 2597-2604.
[5] Jiang N, Wang Z B, Jin Y, et al. Measurement of interface bond quality of composite structure using nonlinear ultrasound. Acta Armamentarh, 2014, 35(3): 398-402.
[6] Yan D W, Simon A N, Bruce W. Modeling and measurement of the nonlinear behavior of kissing bonds in adhesive joints. NDT & E International, 2012, 47(4): 18-25.
[7] Hong M, Su Z H, Wang Q, et al. Modeling nonlinearities of ultrasonic waves for fatigue damage characterization: theory, simulation, and experimental validation. Ultrasonics, 2014, 54(3): 770-778.
[8] Liu S M, Steven B, Simon A N, et al. Measuring bulk material nonlinearity using harmonic generation. NDT & E International, 2012, 48(2): 46-53.
[9] Hirsekorn S. Nonlinear transfer of ultrasound by adhesive joints-a theoretical description. Ultrasonics, 2001, 39(1): 57-68
[10] Hurley D C, Fortunko C M. Determination of the nonlinear ultrasonic parameter using a Michelson interferometer. Measurement Science and Technology, 1997, 8(6): 634-642.
[11] Zhou Z G, Liu S M. Nonlinear ultrasonic techniques used on nondestructive testing: a review. Journal of Mechanical Engineering, 2011, 47(8): 2-11.
[12] Yang S S, Pan Q, Pi W F, et al. Effect of the curing state of the liner on the adhesion properties of the liner and propellant. Chinese Journal of Explosives & Propellants, 2010, 33(3): 88-90.

衬层固化性能的非线性超声检测

江　念[1]，王召巴[2]

(1. 华中光电技术研究所 武汉光电国家研究中心，湖北 武汉 430223；
2. 中北大学 信息与通信工程学院，山西 太原 030051)

摘　要： 基于超声纵波透射法，结合衬层样本的红外光谱图，研究了衬层固化过程中其微观组织结构与超声非线性系数间的关系。利用铌酸锂晶片的滤波特性，在获得最大激励能量的同时有效减少了前端仪器引入的非线性，提高了测量数据的准确性。检测结果表明，在 40 ℃固化温度下，随固化度增加，超声非线性系数先急剧下降而后逐渐减小，与红外光谱技术检测结果一致。因此，利用超声非线性系数可以对衬层的固化性能进行超声无损检测。

关键词： 衬层；固化性能；非线性超声；无损检测

引用格式： JIANG Nian, WANG Zhao-ba. Nonlinear ultrasonic detection for curing properties of the liner. Journal of Measurement Science and Instrumentation, 2018, 9(4): 321-325. [doi: 10.3969/j. issn. 1674-8042. 2018. 04. 003]