Chapter 3 Skin Effect of Acoustic Anisotropy and Dissolved Hydrogen in Metals

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Abstract The work is devoted to study the influence of damaged surface layer on mechanical properties of metal structures. Comparative studies of acoustic anisotropy distributions and dissolved hydrogen concentrations measured according to the vacuum heating method were carried out. The correlation of distributions in rolled steel and aluminum specimens after elasto-plastic and fatigue destruction was revealed. The obtained results indicate the possibility of using the acoustoelasticity method for detecting localized plastic deformations, surface microcracking, and zones with increased concentrations of dissolved hydrogen in metal. It can be used to develop new nondestructive ultrasonic approaches in technical diagnostics of metal structures.

Keywords Acoustic birefringence · Dissolved hydrogen · Elasto-plastic deformation · Nondestructive ultrasonic testing · Vacuum heating method

3.1 Inroduction

The acoustoelasticity method is an ultrasonic nondestructive testing method used to estimate mechanical stresses in solids $[1–7]$ $[1–7]$ $[1–7]$. It is based on phenomenon of acoustic birefringence observed in elastically stressed materials with a crystalline structure. This phenomenon is also called the acoustoelastic effect. It consists in splitting elastic shear waves into components V_1 , V_2 polarized along axes of principal stresses σ_1 , σ_2

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[©] The Author(s), under exclusive license to Springer Nature Switzerland AG 2021 V. A. Polyanskiy and A. K. Belyaev (eds.), *Advances in Hydrogen Embrittlement Study*, Advanced Structured Materials 143, https://doi.org/10.1007/978-3-030-66948-5_3

in case of plane stress state. The acoustoelastic effect was theoretically predicted in 1930s and 1940s by Biot $[8]$ $[8]$, Birch $[9]$, Lazarus $[10]$ $[10]$, and others.

The modern technology of acoustoelastic measurements is based on the theory of wave propagation in a nonlinearly elastic stressed medium. It was obtained after the research was carried out by Hughes and Kelly [\[11\]](#page-8-4), Toupin and Bernstein [\[12](#page-8-5)], Truesdell [\[13](#page-8-6)], Tokuoka and Saito [\[14\]](#page-8-7), Iwashimizu and Kubomura [\[15\]](#page-8-8), Okada [\[16,](#page-8-9) [17\]](#page-8-10), Clark [\[18\]](#page-8-11) and others. Generalization of results obtained for more than fifty years was carried out by Fukuoka in [\[19,](#page-8-12) [20](#page-8-13)].

The essence of results proposed in $[11–17]$ $[11–17]$ is contained in relation (3.1) obtained by Hirao and Pao in [\[21,](#page-8-14) [22\]](#page-8-15) for a weakly anisotropic material:

$$
\frac{V_1 - V_2}{V_0} = \frac{C_{55} - C_{44}}{2\mu} + \frac{4\mu + n}{8\mu^2} (\sigma_1 - \sigma_2) + \alpha_1 (\varepsilon_1^p - \varepsilon_2^p), \tag{3.1}
$$

where $\Delta a = (V_1 - V_2)/V_0$ is non-dimensional parameter of total acoustic anisotropy of material, μ is the second-order Lame constant, *n* is the third-order Murnaghan constant $[23]$, C_{44} and C_{55} are elastic constants in Voigt's notation, α_1 is the exper-imentally determined constant according to Hirao and Pao [\[21,](#page-8-14) [22\]](#page-8-15), σ_1 , σ_2 are the principal biaxial stresses, ε_1^p , ε_2^p are the principal plastic deformations. Nondestructive testing methods based on estimating value of acoustic birefringence widely use the acoustic anisotropy parameter Δa .

Classical acoustoelasticity is limited by the use of first two components in relation [\(3.1\)](#page-1-0). The first component $\alpha_0 = (C_{55} - C_{44})/2\mu$ is related with intrinsic anisotropy caused by microstructure and anisotropic texture of material. The second component $((4\mu + n)/8\mu^2)(\sigma_1 - \sigma_2)$ is related with the acoustoelastic effect in case of an elastically stressed medium. The experimental results of linear acoustoelasticity were obtained by Benson and Raelson [\[24](#page-8-17)], Smith [\[25\]](#page-9-0), Crecraft [\[26](#page-9-1), [27\]](#page-9-2), Hsu [\[28](#page-9-3)], Papadakis [\[29](#page-9-4)], Blinka and Sachse [\[30\]](#page-9-5), Egle and Bray [\[31,](#page-9-6) [32\]](#page-9-7), Kino [\[33](#page-9-8)], King [\[34\]](#page-9-9), Janssen [\[35](#page-9-10)] and others.

Hirao and Pao [\[21,](#page-8-14) [22](#page-8-15)] proposed to expand application of the acoustoelasticity method [\[24\]](#page-8-17) by including component $\alpha_1(\epsilon_1^p - \epsilon_2^p)$ linearly related to plastic deformations [\(3.1\)](#page-1-0). It was experimentally verified in the case of four-point bending of annealed prismatic beams at plastic deformations of 1% order [\[21\]](#page-8-14). The study of small plastic deformations according to relation (3.1) was also carried out in [\[36](#page-9-11)].

More than 30 years have passed since the appearance of [\[21,](#page-8-14) [22\]](#page-8-15) results. Recent studies indicate a significant influence of damage accumulated during monotonic [\[37\]](#page-9-12) and cyclic loading [\[38](#page-9-13)] on acoustic anisotropy of industrial structures. In addition, influence of hydrogen embrittlement on ultrasonic measurements was found during standard hydrogen-induced corrosion tests (HIC) [\[39\]](#page-9-14).

At the same time, uneven accumulation of plastic deformation and damage is usually observed. The periodic structure of plastic deformation waves called "chessboard" is observed in polycrystalline materials [\[40](#page-9-15)[–42\]](#page-9-16). Microcracks localized in a thin surface layer have a significant influence on the degradation of mechanical properties of metal [\[43](#page-9-17), [44\]](#page-9-18). Therefore, the use of relation [\(3.1\)](#page-1-0) obtained for specially prepared isotropic specimens may be incorrect for real structures.

The aim of this work is to carry out comparative studies of acoustic anisotropy and high-precision measurements of hydrogen concentrations in rolled specimens from steel and aluminum alloys.

3.2 Experiments

3.2.1 Methods

Three stages of experimental research were carried out. At the first stage, metal specimens cut from rolled sheet were subjected to uniaxial loading. Thus, the Hirao– Pao relationship [\(3.1\)](#page-1-0) was tested in a wide range of loads and deformations.

At the second stage, ultrasonic measurements were carried out using the Benson– Raelson approach [\[24\]](#page-8-17) with calculation of total acoustic anisotropy Δa according to relation [\(3.2\)](#page-2-0):

$$
\Delta a = \frac{V_1 - V_2}{(V_1 + V_2)/2},\tag{3.2}
$$

where denominator $(V_1 + V_2)/2$ is used instead of shear wave velocity V_0 for an unstressed isotropic material.

Ultrasonic measurements were carried out using a standard sensor with 5MHz acoustic signal frequency. Wave packets were emitted and received by 12×12 mm piezoelectric transducers. The ultrasonic sensor was controlled by an acoustic anisotropy analyzer [\[5](#page-8-18)]. This device allowed one to obtain average time delays t_1 , t_2 between multiple reflected pulses. The velocities V_1 , V_2 were obtained after direct micrometric measurement of specimen thickness *h*. Shear waves were polarized along and perpendicular to direction of uniaxial loading.

At the third stage, hydrogen concentrations were measured by the vacuum heating method [\[45,](#page-10-0) [46](#page-10-1)]. It is based on use of hydrogen diffusion during heating of analyzed metal. The specimen extraction curves contain dependence of extracted hydrogen flux on heating time in vacuum.

Dissolved hydrogen measurements were carried out using high-precision industrial analyzer. Small prismatic specimens with 8 mm-size were tested. The experimental technique for determining hydrogen concentrations is described in [\[47](#page-10-2)].

3.2.2 Acoustoelastic Effect in Case of Uniaxial Elasto-Plastic Deformation

The total acoustic anisotropy Δa and velocities of ultrasonic shear waves V_1 , V_2 in the case of uniaxial elasto-plastic tension were investigated. Cold-rolled 500×70 \times 15 mm specimens from AMts aluminum alloy were tested. Uniaxial rigid loading was carried out with 5 mm step on INSTRON-8806 hydraulic tensile testing machine.

One of the specimens after destruction and unloading is shown in Fig. [3.1.](#page-3-0) It was subjected to 19 loading stages. Acoustic measurements were carried out at 10 points located along the specimen axis (in Fig. [3.2\)](#page-3-1). The total axial deformation ε up to specimen destruction was equal to 26.70%. The initial distribution of acoustic anisotropy Δa obtained along specimen axis turned out to be close to uniform and was equal to $a_0 = 0.50 \pm 0.03\%$.

Measurement points No. 1, 4, 7, and 10 were located at 45, 90, 135- and 180-mm distance from destruction zone. The results of acoustic anisotropy measurements at each loading stage are shown in Fig. [3.3.](#page-4-0)

3.2.3 Comparative Studies of Acoustic Anisotropy and Hydrogen Concentrations

Comparative studies of acoustic anisotropy distributions Δa , % and hydrogen concentrations *CH*, ppm were carried out at $n = 10$ points located along specimen axis (see Fig. [3.2\)](#page-3-1). The hydrogen concentrations were investigated including surface layer and inside the aluminum specimen at 2 mm depth from its surface. The obtained results are shown in Figs. [3.4](#page-4-1) and [3.5.](#page-4-2)

Also, studies on rolled COR-TEN steel specimen (in Fig. [3.6\)](#page-5-0) were carried out. The specimen was subjected to 380 kN cyclic load and destroyed after $n = 5 \times 10^6$ loading cycles (in Fig. [3.7\)](#page-5-1).

Similar studies of acoustic anisotropy Δa , % and dissolved hydrogen concentrations *CH*, ppm were carried out at $n = 10$ points located along steel specimen axis. The results obtained near fatigue destruction zone are shown in Figs. [3.8](#page-5-2) and [3.9.](#page-5-3)

3.3 Discussion

3.3.1 Nonlinear Dependence of Acoustic Anisotropy During Elasto-Plastic Deformation

Several important conclusions follow from results presented in Fig. [3.3.](#page-4-0) The contribution of elastic deformations to the value of acoustic anisotropy averaged 0.54%. It is comparable to initial acoustic anisotropy due to influence of anisotropic texture. Thus, the contribution of component a_0 must be studied during acoustoelastic measurements in cold-rolled structures.

The dependence of acoustic anisotropy is nonlinear with increasing deformations (see Fig. [3.3\)](#page-4-0). All experimental curves have non-monotonic region at deformations equal to $\varepsilon = 1.42 \div 2.82\%$. It is observed with the beginning of plastic flow process.

Two other non-monotonic regions are observed for points No. 1 and 4 after deformations equal to $\varepsilon = 19.20\%$. Significant changes in acoustic anisotropy value are related with the specimen surface curvature due to plastic neck formation. Semenov [\[37,](#page-9-12) [43\]](#page-9-17) obtained relation [\(3.3\)](#page-6-0) for a small deviation α angle from the surface normal. In this case, acoustic anisotropy Δa is calculated as

$$
\Delta a = \alpha^2 \frac{9(\lambda + \mu)}{(4(\lambda + \mu) - r)} \frac{r}{\mu},
$$
\n(3.3)

where λ , μ are the Lame constants, $r = 4\mu^2/(H' + 3\mu)$, H' is inclination angle of stress–strain diagram [\[37,](#page-9-12) [43](#page-9-17)].

The actual dependence of acoustic anisotropy on plastic deformations has a nonlinear nonmonotonic character (see Fig. [3.3\)](#page-4-0) and cannot be described by linear dependence $\alpha_1(\varepsilon_1^p - \varepsilon_2^p)$ from relation [\(3.1\)](#page-1-0). It differs from previously published results obtained on basis of the Murnaghan's theory of finite deformations [\[23\]](#page-8-16). Alternative mathematical models [\[37](#page-9-12)] should be used to describe propagation of shear waves at large plastic deformations of material.

3.3.2 Skin Effect of Acoustic Anisotropy and Hydrogen Concentrations

The formation of metal defects in atmosphere not subject to drying or filling with inert gases is related to an increase in hydrogen concentrations. Hydrogen is an indicator of structural changes in metals such as cracking, loosening, pore formation, and others. It also leads to formation of new defects under external loading.

The diffusion of hydrogen has a great influence on processes of corrosion, cracking, and brittle fracture of metals. The mechanism of hydrogen embrittlement was explained by Gorsky [\[48\]](#page-10-3). He found that deformations of crystalline matrix caused by mechanical stresses have an influence on hydrogen diffusion in solids.

Comparative studies of hydrogen concentrations and acoustic anisotropy distributions revealed their correlation both on aluminum (in Figs. [3.4](#page-4-1) and [3.5\)](#page-4-2) and steel specimens (in Figs. [3.8](#page-5-2) and [3.9\)](#page-5-3). This effect cannot be due to residual stresses or influence of crystal structure. The observed macroscopic distributions significantly exceed the effects related with plastic flow, such as formation of Luders bands or the Portevin-Le Chatelier effect [\[49\]](#page-10-4). It is similar to the phenomenon of propagation of plastic flow autowaves discovered by Zuev [\[50\]](#page-10-5).

At the same time, the qualitative coincidence of ultrasonic and hydrogen results can be due to influence of microcracks leading to material loosening under monotonic or cyclic load. The uniform distribution of hydrogen inside the specimen (in Fig. [3.5\)](#page-4-2) indicates the surface effect of hydrogen accumulation [\[44\]](#page-9-18). It also indirectly indicates the surface effect of acoustic anisotropy predicted earlier in [\[43\]](#page-9-17).

3.4 Conclusions

Experimental studies have revealed nonlinear non-monotonic character of the acoustic anisotropy dependence on elasto-plastic deformations for orthotropic specimens. This effect is related with the beginning of different stages of deformation process. It cannot be described using generally accepted acoustoelasticity relations postulating linear dependence on plastic deformations. Thus, the influence of large plastic deformations on shear wave velocities should be described using alternative models not based on Murnaghan's theory of finite deformations.

The revealed correlation between acoustic anisotropy distributions and dissolved hydrogen concentrations can be explained only by formation of microcracks systems localized in a thin surface layer of metal. The results of comparative ultrasonic and hydrogen studies indicate the possibility of detecting metal saturation with hydrogen using acoustic anisotropy measurements.

The obtained results allow one to apply the acoustoelasticity method to nondestructive testing of plastic deformations and microcracking of metal structures at any stage of their operation.

Acknowledgements The research is carried out under the financial support by Russian Science Foundation, project 18-19-00413.

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