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DEVELOPMENT OF HIGH-RESOLUTION MECHANICAL SPECTROSCOPY, HRMS: STATUS AND PERSPECTIVES. HRMS COUPLED WITH A LASER DILATOMETER

POWSTANIE I ROZWÓJ WYSOKOROZDZIELCZEJ SPEKTROSKOPII MECHANICZNEJ HRMS - STAN OBECNY I PERSPEKTYWY. HRMS SPRZĘŻONY Z DYLATOMETREM LASEROWYM

Recent achievements in the development of low-frequency high-resolution mechanical spectroscopy (HRMS) are briefly reported. It is demonstrated that extremely low values of the loss angle, φ, ($\tan\varphi_b = 1\times10^{-5}$) can be measured as a function of frequency, and the precision in estimation of the dynamic modulus is better than 1×10^{-5} in arbitrary units. Three conditions must be fulfilled to obtain high resolution in subresonant and resonant mechanical loss measurements: (1) noise in stress and elastic strain signals must be lower than 70 dB, (2) high quality of stress and strain signals must be tested both in the frequency- and time-domains, and (3) the estimation of the mechanical loss and modulus must be verified by at least two different computing methods operating in the frequency- and time-domains. It is concluded that phase measurements in the subresonant domain are no longer determined by precision in estimation of the loss angle. Recent developments in high-resolution resonant mechanical loss measurements stem from the application of advanced nonparametric and parametric computing methods and algorithms to estimate the logarithmic decrement and the elastic modulus from exponentially damped free decaying oscillations embedded in experimental noise.

It is emphasized that HRMS takes into account the presence of noise in the stress and strain signals, which has not yet been addressed in the literature. The coupling of a low-frequency mechanical spectrometer with an *in-situ* laser dilatometer is suggested as a new perspective research area in Materials Science.

Keywords: mechanical spectroscopy, internal friction, elastic modulus, noise, Fourier transform, Hilbert transform, joint-time frequency analysis, dilatometry

W pracy przedstawiono najnowsze osiągnięcia związane z powstaniem i rozwojem niskoczęstotliwościowej wysokorozdzielczej spektroskopii mechanicznej, HRMS. Wykazano, że możliwym jest pomiar skrajnie niskich wartości kąta strat φ, ($\tan\varphi_b = 1\times10^{-5}$) mierzonych w funkcji częstotliwości, zaś dokładność pomiaru dynamicznego modułu sprężystości jest lepsza niż 1×10^{-5}, w jednostkach względnych. Do uzyskania wysokiej rozdzielczości w zakresie subrezonansowej i rezonansowej spektroskopii mechanicznej koniecznym jest spełnienie trzech warunków: (1) szum w sygnałach naprężeń i odkształceń sprężystych musi być bardzo niski, tzn. poniżej poziomu 70 dB, (2) sygnały naprężeń i odkształceń sprężystych muszą być wysokiej jakości i muszą przejść stosowne testy zarówno w dziedzinie częstotliwości, jak i czasu, (3) obliczone wartości strat mechanicznych i modułu sprężystości muszą być zweryfikowane przez co najmniej dwie różne metody obliczeń prowadzone w dziedzinie częstotliwości i czasu. Jednym z najważniejszych wniosków jest stwierdzenie, że pomiary różnic w fazie pomiędzy sygnałami naprężenia i odkształcenia sprężystego w zakresie subrezonansowym, nie są zdeterminowane, jak dotychczas twierdzono, przez ograniczenia w dokładności obliczeń kąta strat. Najnowsze osiągnięcia uzyskane w rozwoju wysokorozdzielczej rezonansowej spektroskopii mechanicznej wynikają z zastosowania nowych zaawansowanych nieparametrycznych i parametrycznych metod obliczeń i algorytmów do estymacji logarytmicznego dekrementu tłumienia oraz modułu sprężystości z wykładniczo tłumionych swobodnie zanikających oscylacji zawierających szum eksperymentalny.

W pracy podkreślono, że wysokorozdzielcza spektroskopia mechaniczna HRMS uwzględnia obecność szumu w sygnałach naprężeń i odkształceń, która dotychczas nie była brana pod uwagę w literaturze światowej. Wykazano również, że połączenie niskoczęstotliwościowego spektrometru mechanicznego z in-situ dylatometrem laserowym w jednym urządzeniu badawczym otwiera nowe możliwości poznawcze w zakresie inżynierii materiałowej.

1. Fundamentals of high-resolution mechanical spectroscopy, HRMS

High-resolution mechanical spectroscopy (HRMS) is a new term introduced in 2012 [1-4] for new methods of computing the logarithmic decrement, δ, and the resonant frequency, f_0, as a function of several experimental parameters and noise, which is inherent in discrete signals of exponentially damped time-invariant harmonic oscillations (free-elastic decaying signals) [1-9] and in time-invariant harmonic forced

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oscillations. The HRMS program was designed to develop novel research capabilities in subresonant [10-15] (forced harmonic oscillations) and resonant domains [1-9, 15-18] (free decaying oscillations).

The program for the subresonant domain is comprised of: (1) precise measurements and analysis of stress and strain signals recorded in a low-frequency forced mechanical spectrometer; (2) measurements and computations of the mechanical loss angle, φ (i.e. the mechanical loss tangent, $\tan\varphi$, where φ denotes the phase angle between stress and strain signals); (3) measurements of very low internal friction background, measured as a function of the excitation frequency in the range from 0.0001 to 1 Hz; (4) precise estimation of the dynamic modulus.

The HRMS program for the resonant domain comprises advanced computation methods relating to the logarithmic decrement, δ, and the resonant frequency, f_0, which are insensitive to the detrimental zero-point drift (ZPD) effect and offset [8], and robust against experimental noise and other undesirable nonharmonic perturbations [1-9].

The interpretation of mechanical loss spectra in solids is often impeded by: (1) complexity of overlapping peaks, (2) the presence of marginally discernable internal friction peaks, (3) the presence of the ZPD, (4) very large scatter in the experimental data, (5) poorly resolved background on the low-temperature (*or* low-frequency) and/or the high-temperature (*or* high-frequency) side of internal friction peaks, (6) transient effects, and (7) nonequilibrium conditions when making the measurements. These effects may not only cause serious experimental problems, but may also make the interpretation of internal friction peaks devoid of physical meaning. These problems can be greatly alleviated by nonparametric computation methods of the logarithmic decrement and elastic modulus, as discussed in Section 6.

The development of low-frequency mechanical spectroscopy of metals and alloys is hindered by experimental limitations listed in Table 1. The majority of these problems are solved in this work.

HRMS has emerged from a number of technical advances that include generation of high-quality stress signals (torque), detection of high-quality time-dependent elastic strain signals (angular displacement), electronics, including analog to digital converters, signal processing, automated data collection, and importantly, computational methods. The latter are of crucial importance for the estimation of: (1) the loss tangent, $\tan\varphi$, and the dynamic shear modulus, G, and (2) the logarithmic decrement, δ, and the resonant frequency, f_0.

The uniqueness of our HRMS facilities consists in the use of a laser triangulation system to measure harmonic strain signal characterized by low noise (signal-to-noise ratio (S/N), S/N = 70 dB), high-quality control of the driving system (stress signal), and a matured design. These advances impart new qualities to the HRMS experimental results. This paper demonstrates that the application of new solutions to measure high-quality stress and strain signals with negligible noise gives important information pertaining to the technical requirements of HRMS. The following issues are discussed: internal friction background, the resolution limit in estimation of the modulus, and the quality of the stress and strain signals. Furthermore, other parameters that determine the quality of mechanical loss measurements are considered.

TABLE 1

Critical experimental limitations in low-frequency mechanical spectroscopy

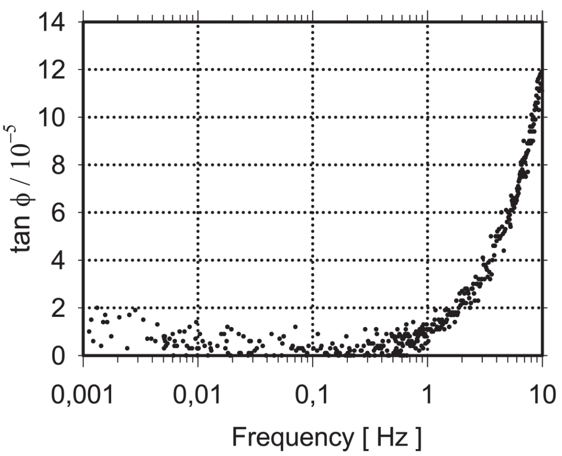
Subresonant domain. Forced harmonic oscillations	
1. High background in measurements of the loss tangent, $\tan\varphi$	$\tan\varphi_b$ from 4×10^{-3} to 1×10^{-2} [DMA instruments] $\tan\varphi_b \approx 1\times10^{-3}$ [Kê-type forced torsion pendulum] High resolution: $\tan\varphi_b = 1\times10^{-5}$ [this work]
2. Dispersion in the loss tangent, $\tan\varphi$	Dispersion in values of the $\tan\varphi$ below the level ~2×10^{-3} High resolution: $\tan\varphi = \pm 1\times10^{-5}$ [this work]
3. Resolution in the shear dynamic modulus G (in arbitrary units)	Dispersion in the shear dynamic modulus, G High resolution: G = $\pm 1\times10^{-5}$ at 0.01 Hz [this work] G = $\pm 5\times10^{-6}$ at 0.10 Hz [this work]
4. Low density of experimental points. 5. Long experimentation time.	
Resonant domain. Exponentially damped harmonic oscillations	
1. Dispersion in the logarithmic decrement, δ	Low damping: dispersion in the values of internal friction, Q^{-1}, below 5×10^{-4}
2. Dispersion in the logarithmic decrement, δ	High damping: dispersion in the values of internal friction, Q^{-1}, above 0.01
3. Low density of experimental points to reveal sharp and narrow internal friction peaks.	

2. Subresonant domain. Internal friction background

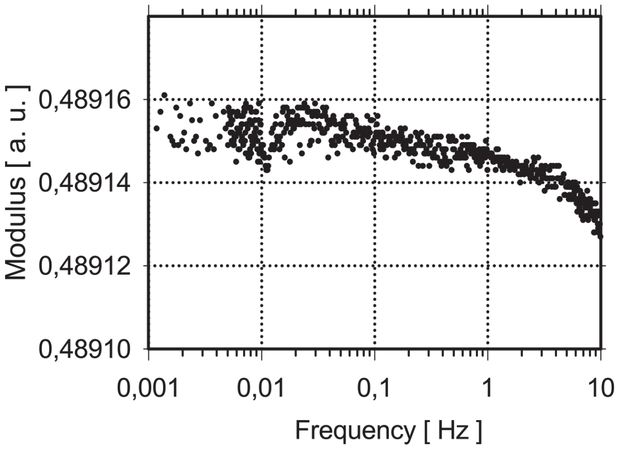
Until now, the measurements of the mechanical loss angle, φ, in metals and alloys in forced oscillations has been a tedious chore, producing less than desirable results. A widespread opinion is that the major drawback of low-frequency subresonant mechanical spectrometers is an excessive level of the internal friction background, $\tan\varphi_b \approx 0.001$ (Table 1). It is believed that such a small loss angle between the leading stress and the lagging strain is comparable to the precision of phase measurement.

Figure 1a indicates that a much smaller loss tangent, $\tan\varphi_b = 1\times10^{-5}$ (background level in tantalum), can be experimentally measured in the frequency range 1×10^{-3} to 0.9 Hz. Therefore it can be concluded that internal friction background in forced oscillations is not determined by the precision of the phase measurement. This is why other parameters affecting the background should be reconsidered: (1) the quality of the driving stress and resulting strain signals, (2) the level of noise in stress and strain signals, (3) the presence of undesirable parasitic harmonic components (with low amplitudes), (4) undesirable external electromagnetic and mechanical perturbations, (5) signal processing parameters, (6) algorithms to estimate the phase lag, (7) the use of permanent magnet and coil designs which typically introduce thermal drift into the system, (8) long-term drift, (9) external friction induced by sample clamping or magneto-mechanical damping (in ferromagnetic samples). Hence it can be assumed that the 'true internal friction background', $\tan\varphi_b$, stems from dissipation of mechanical energy in a sample. The electronic background in the HRMS spectrometer is below 4×10^{-7} in the frequency range 1×10^{-3} to 10 Hz.

Figures 1a and 2a show the effect of the number of oscillations (the number of periods), Nosc, on estimation of $\tan\varphi$ under the same experimental conditions. Figure 1a was obtained for the optimized number of oscillations. In contrast, Fig. 2a indicates the effect of a small number of oscillations, Nosc = 3, on the loss tangent (three oscillations are used in commercially available frequency analyzers), brought about a large dispersion of experimental points.

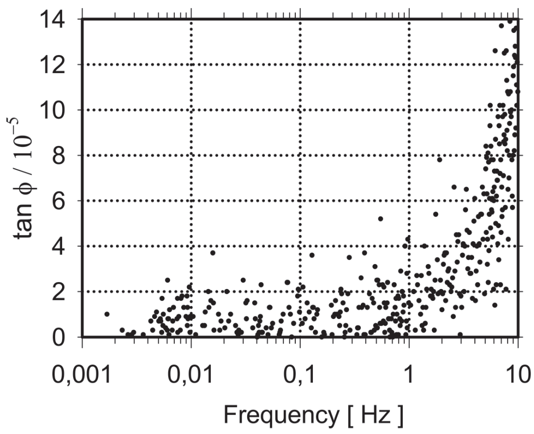


(a)

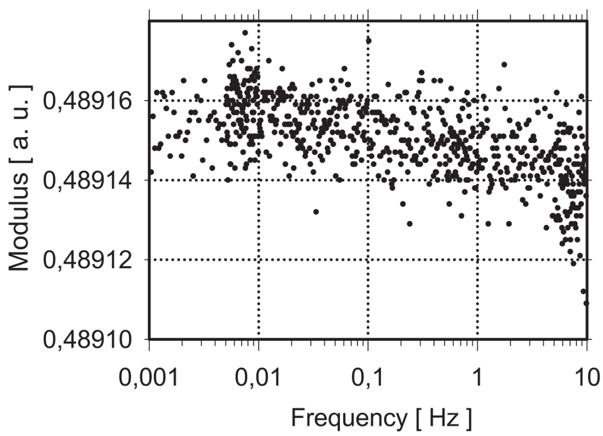


(b)

Fig. 1. The performance of the high-resolution mechanical spectrometer, HRMS. Variation of the tan φ (a) and modulus (b) with frequency for the optimized number of oscillations, Nosc. The frequency sweep was measured in the range from 10 to 10^{-3} Hz. The signal-to-noise ratio S/N = 70 dB



(a)



(b)

Fig. 2. The performance of the high-resolution mechanical spectrometer, HRMS. Variation of the tan φ (a) and modulus (b) with frequency for a small number of oscillations, Nosc = 3. The frequency sweep was measured in the range from 10 to 10-3 Hz. The signal-to-noise ratio S/N = 70 dB

It is important to recall that the internal friction background in subresonant low-frequency mechanical spectrometers is affected by the natural frequency, f_n, of the system. At sufficiently low driving frequency, the measured loss angle, $\tan\varphi_m$, becomes equal to the mechanical loss angle, $\tan\varphi_a$. The correction factor is given by:

$$\tan\varphi_a = \tan\varphi_m\left[1-\left(\frac{f}{f_n}\right)^2\right]. \quad (1)$$

For a metallic sample and the natural frequency f_n = 100 Hz, the error obtained at the excitation frequency is around 1%. This error decreases by two orders of magnitude when the excitation frequency increases by one order of magnitude. Thus, at 1 Hz the error is around 0.01%.

The effect of the natural frequency, f_n, of the system on dynamic modulus has not been yet established.

3. Subresonant domain. Modulus

Figure 1b presents the values of the modulus measured in our high-resolution facilities as a function of the excitation (driving) frequency. As shown, remarkably, the changes in the modulus were detected with a resolution of 1×10^{-5}, better by at least one order of magnitude than typically reported. Figure 2b illustrates the results obtained when an inadequate number of oscillations (periods) (Nosc = 3) was used. This leads to the visible dispersion of the experimental points. From these data, it follows that the resolution of the results improved when the number of oscillations reached a certain limit.

It should be clearly stated that the indispensable condition for obtaining high-resolution measurements of both the $\tan\varphi$ (Fig. 1a) and the modulus (Fig. 1b) is the reduction of the noise that can be of different origins, such as electrical or external electromagnetic. In this work, the noise was brought down to as low a level as S/N = 70-85 dB.

The high-resolution technique is attractive as a quantitative method of studying dislocation-point defect interactions, pinning stages, pre-transformation phenomena, phase transitions, annealing kinetics, and asymmetries of internal friction relaxation peaks in metals, alloys and other solids [16,17,19-21]. The estimate of the dynamic modulus obtained from discrete Fourier transform of stress and strain signals (Fig. 1a) satisfies HRMS technical requirements. The modulus curve deviates slightly from linearity at frequencies above 2 Hz (Fig. 1b). The reason for this small deviation is unknown and requires further studies of the vibration system (mechanical spectrometer). Nevertheless, the dispersion of the modulus experimental points obtained in this work is far lower than those reported in the literature (compare the scale in the Y-axis in Figs. 1b, 2b).

The problem of dispersion of the modulus points observed in forced oscillations has received scant attention to date. The scatter of experimental points and unusual modulus variation as a function of excitation frequency reported for martensitic carbon steels [22,23] should be reinterpreted with noise taken into consideration.

In conclusion, it is worthwhile to emphasize that Fig. 1b demonstrates excellent estimation of dynamic modulus. The results suggest what can be achieved, provided the quality of the harmonic stress and strain signals is sufficiently high and time-invariant.

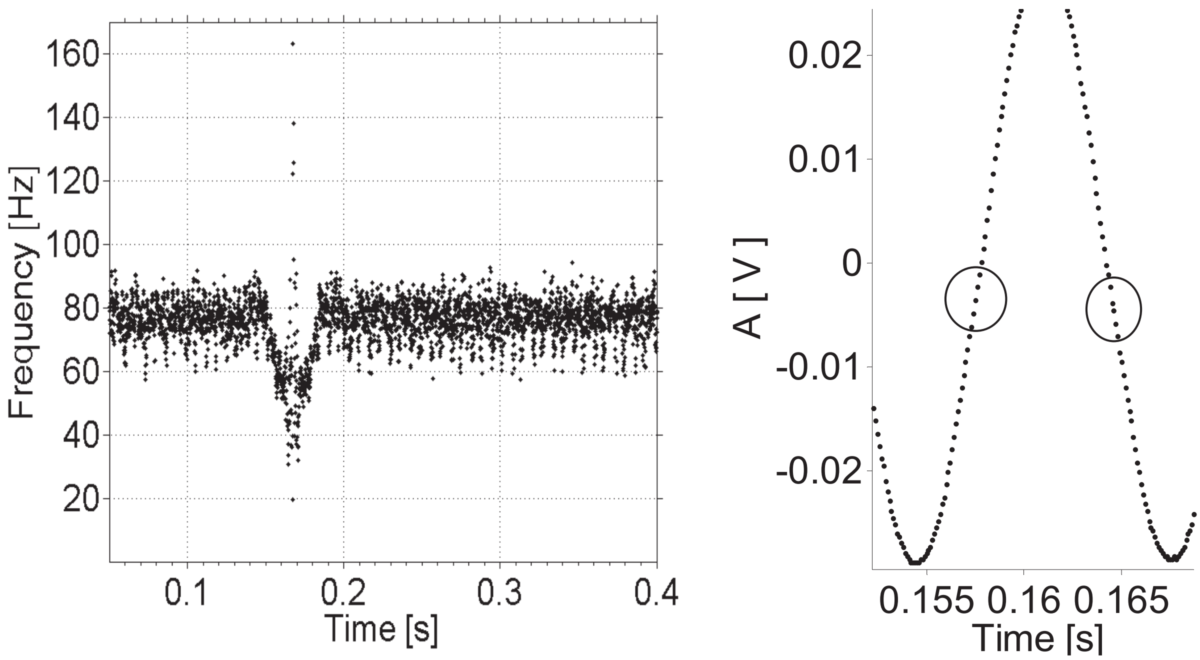
4. Signal quality test of stress and strain signals

The high-resolution mechanical spectrometer is a novel instrument that serves two functions. It is not only an instrument to measure the loss of mechanical energy and the dynamic modulus in solids, but also a tool for on-line control of stress and strain signals in time- and frequency-domains. In the former, the following computing methods can be used: (1) the parametric Optimization in Multiple Intervals (OMI) method [1-9], (2) the Hilbert-twin (H-twin) method [9], and (3) numerous classical methods [5-8]. In the latter, the stress and strain signals can be analyzed by discrete Fourier transform (DFT) [5-7] and interpolated discrete Fourier transform (IpDFT) [1-4,9].

The Fourier transform provides the power spectrum, which reveals the presence of parasitic components at distinct frequencies (different from the main torsion component). The Fourier transform, however, has a global character and is not appropriate for characterization of the local signal parameters (i.e. stress and strain signals in resonant and subresonant mechanical spectrometers) [24-27]. The high-resolution time-domain analysis and the time-frequency representation of signals [28] (see Fig. 3) are powerful tools in a signal quality test of stress and strain signals recorded in a mechanical spectrometer. These tests can provide a wealth of information concerning the quality of excitation stress, lagging strain and free-elastic decaying signals.

To check the effectiveness of the aforementioned joint time-frequency representation, it was applied for the analysis of free decaying oscillations. The oscillations were recorded in another laboratory (in the framework of a round robin test) with a laser instrument ILD from Micro-Epsilon. The results of the analysis are presented in Fig. 3. The analysis revealed that the free decaying oscillations had not been recorded at equal time intervals (such a detail could never have been detected with Fourier spectral analysis). As a result of the above, a characteristic spike was formed, Fig. 3a. The spike indicates that the values of both the logarithmic decrement and the resonant frequency estimated with the interpolated discrete Fourier transform (the classic Yoshida method (Y) [29], the Yoshida-Magalas method (YM) [1-4,9]) and the Hilbert-twin (H-twin) method [9] are bound to be negatively affected. The reason is straightforward, in that the integral transform methods developed herein to compute δ and f_0, as well as the $\tan\varphi$ and the modulus, can be applied only if the digitized data are linear and strictly periodic or stationary. This condition was not fulfilled by the ILD instrument used in the partner laboratory (Fig. 3b).

It should be emphasized that the traditional classical approach [16,17] to the estimation of the logarithmic decrement based on classical methods [5,15-17] does not give a true description of the dissipation of mechanical energy for nonlinear damping phenomena, amplitude-dependent internal friction and non-exponentially damped harmonic oscillations.



(a) (b)

Fig. 3. The signal quality test of free decaying oscillations: $\delta = 1.51 \times 10^{-2}$, the resonant frequency $f_0 \approx 76.39$ Hz, the sampling frequency f_s = 10 kHz. (a) The time-frequency analysis yields a spike, which precisely localizes irregular sampling time-interval recorded by the ILD Micro-Epsilon instrument. (b) A zoom on the analyzed free decaying oscillations indicates an irregular sampling time-interval

5. Effect of noise

The importance of noise in strain and stress signals has not been dealt with in the literature devoted to internal friction and mechanical spectroscopy - except for the data reported in Refs. [1-9]. Noise, indiscernible when improper sampling frequencies f_s are applied, always affects the results obtained with different methods and algorithms [1-9]. In our previous reports [5,6] we demonstrated that an increase in noise level in free-elastic decaying oscillations increases the dispersion in the logarithmic decrement, δ, and in the resonant frequency, f_0 . This is why the concept of exponentially damped time-invariant harmonic oscillations embedded in experimental noise, ε_w(t), was defined as the '**free-elastic decay**' [9]. This definition allows a clear distinction to be made between the 'free decay' and the 'free-elastic decay'. While in the former, noise is tacitly assumed to be absent, in the latter it appears in the experimental data. Thus it is implicit that noise must be taken into account in the HRMS and the computational methods developed must be robust against experimental noise.

Obviously, additive and multiplicative noise [30] in the HRMS must be as small as possible. In this work the S/N was improved from 38-60 dB [1-9] to 85 dB. This was achieved by developing a new low-noise strain detection system, appropriate hardware solutions, and signal processing. It is noteworthy to mention that very low sampling frequencies, f_s , yield higher dispersion in the δ (see Fig. 3 in [5] and Figs. 1-8 in [3]) and in the f_0 experimental points (see Fig. 3 in [7] and Figs. 1-4 in [2].)

To quantitatively describe exponentially damped harmonic oscillations embedded in experimental noise is a mathematically challenging problem. Several classical methods to compute the logarithmic decrement were reported in Refs. [5-8]. The Fourier and Hilbert transform-based methods advocated for the computation of δ and f_0 were successfully tested for different noise levels [1-9]. These innovative methods are tailored to address several issues covering both fundamental understanding of mechanical loss measurements in solids, and realization of hardware and software control in HRMS.

6. New computing methods developed for logarithmic decrement and dynamic elastic modulus

In this work, a high-resolution mechanical spectrometer was developed. For the instrument to be effective, the following computing methods were designed: the parametric Optimization in Multiple Intervals (OMI) method [1-9], the Yoshida-Magalas (YM) method [1-4,9] and the most recent Hilbert-twin (H-twin) method based on the Hilbert transform [9]. The relative effectiveness of these methods is summarized in [9]. These methods are computationally compact and efficient, the OMI being termed the 'gold standard' for the computations of the logarithmic decrement, δ, and the resonant frequency, f_0 [1-4]. While the OMI and YM methods yield the highest resolutions available for δ and f_0 , the Hilbert-twin method provides excellent estimation of the logarithmic decrement, δ [9] obtained from the '**true envelope**' of free-elastic decaying signals. The '**true envelope**' is obtained from the 'twinning procedure', which eliminates effectively ripples, that is, intrinsic asymmetrical oscillations of the envelope obtained from conventional discrete Hilbert transform of free-elastic decaying oscillations [9].

The nonparametric YM method is a computationally efficient, robust and reliable tool to estimate the logarithmic decrement and the resonant frequency from exponentially damped free-elastic decaying signals, including also free decaying signals affected negatively by the ZPD and offset. It is well known that all the interpolated discrete Fourier transform-based methods exhibit an intrinsic limitation due to the spectral leakage effect, which cannot be eliminated by application of various time windows. These constraints limit the resolution in the estimation of the logarithmic decrement for experimental signals acquired in low frequency mechanical spectrometers. The performance of the advocated computing methods is corroborated by simulations and direct comparison between different computing methods, including the classical methods [1-9]. Several computing methods and algorithms designed for the analysis of exponentially damped harmonic oscillations embedded in noise are available in the Free Decay Master Software Package [31].

It is important to point out that numerous tests devoted to the application of a variety of time windows [24-27] do not provide a reliable improvement in estimation of logarithmic decrement and resonant frequency in experimental mechanical loss measurements in the low- and medium-frequency range of vibration (from 0.1 Hz to 70 kHz) for extremely low-, low- (from $Q^{-1} \approx 10^{-8}$ to $Q^{-1} \approx 10^{-4}$), and high-damping levels (Q^{-1} above 0.01.)

It is interesting to note that an uncertainty analysis of computation of the logarithmic decrement was recently described in terms of the uncertainty of damped harmonic signal and the length of free decaying signals (viz. the number of oscillations *or* the number of periods) [32]. This approach is appropriate for computations of the logarithmic decrement according to the oldest and simplest classical method based on counting the number of free decaying oscillations [5,8,16,17].

7. Effect of the zero-point drift, ZPD

The zero-point drift (ZPD) is defined as movement (deployment) of the center of free decaying oscillations recorded in a mechanical spectrometer (e.g. inverted torsion pendulum), that is an unpredictable short-term drift, which is inherent in a free decaying signal and estimation of the logarithmic decrement [8]. ZPD is known to produce a detrimental effect on experimental data acquired during mechanical loss measurements [8]. The presence of ZPD distorts the shape and the symmetry of internal friction peaks, affects peak's temperature and consequently influences the relaxation parameters of internal friction peaks. In the worst case, it may also cause an occurrence of an artificial 'ghost internal friction peak' [8]. Given that classical computation methods applied in mechanical spectroscopy and internal friction measurements are vulnerable to ZPD, it is indispensable that quantitative information on the variation of ZPD during loss measurements and on its impact on estimation of δ , $\tan\varphi$, and modulus is reported. Also, importantly, it was shown that the classical computational methods [5] are inadequate in the presence of ZPD and offset [8]. To alleviate incorrect estimation of δ, it is proposed to use YM or OMI methods [1-9]. It should be remembered, however, that interpolated discrete Fourier transform-based methods (YM and Y methods) are less precise for free decaying signals that are very short [1-4,9].

In view of the above knowledge, the early experimental results reported in the literature, such as Hasiguti peaks observed in freshly deformed Al, Cu, and other metallic samples [16,17,20], should be reinterpreted as being strongly biased by the ZPD effect. This effect also negatively impacts on measurements of internal friction peaks induced by: (1) phase transformations in solids, (2) thermoelastic and non-thermoelastic martensites, (3) plastic deformation of metals and alloys (especially after low-temperature in-situ deformation carried out inside an inverted torsion pendulum and after in-situ deformations at room temperature), (4) quenching of iron-based alloys, that is, during measurements of steel samples with evolving and/or thermodynamically unstable microstructure (e.g. samples containing twinned plate martensite, fresh plate and lath martensite, and/or bainitic structure in Fe-C and Fe-Ni-C alloys, etc.) [22,23,33-43], (5) release of internal stresses in a sample.

To summarize, ZPD poses a major problem for free decaying oscillations, as, in the end, it has an adverse impact on the calculation of δ and the elastic modulus. It is worthwhile to reiterate that numerous examples of inevitable ZPD effect can be found in metals and alloys as a result of evolution of thermally unstable microstructure induced e.g. by phase transformations, plastic deformation, irradiation, quenching, annealing, and time-evolution of other physical properties.

Thus, detrimental effects of ZPD can now be efficiently eliminated by YM and OMI methods, even for free-elastic decaying signals of short duration [9]. Elimination of the ZPD effect in computations of the logarithmic decrement and dynamic elastic modulus is a condition *sine qua non* in the HRMS. It is critically important to emphasize that special care must be taken in conventional mechanical loss measurements (e.g. internal friction measurements performed in an inverted torsion pendulum and other instruments operating in torsion and/or bending) to avoid detrimental effect of the zero-point drift and offset.

8. A round-robin test of the uncertainty of measurements of logarithmic decrement and elastic modulus. Validation of computing methods and algorithms

To ensure reliability of mechanical loss measurements, a round-robin test involving several partner laboratories is recommended. It enables a comparison to be made of various computation methods of estimating internal friction and elastic modulus. This project can be supported by the 'Free-Decay Master Software Program' [31] to analyze various raw experimental data containing different experimental noise, offset, ZPD effect and other non-harmonic disturbances. A round-robin test may also include signal quality tests performed in the time- and the frequency-domains.

9. New perspectives. Low-frequency mechanical spectrometer coupled with a laser dilatometer

Figure 4 shows internal friction peak at 350 K in Fe-C alloy (150 at.ppm of carbon atoms in solid solution) after 4% cold-work at 77 K. Prior to low-temperature deformation, the sample was plastically deformed at room temperature and annealed during the first run-up at 670 K. The 350 K peak is observed only after low-temperature plastic deformation at 77 K. It is important to note that the peak at 350 K is accompanied by a noticeable longitudinal elongation of the sample, detected by a laser triangulation technique. The coupling of a low-frequency mechanical spectrometer with an *in-situ* laser dilatometer is a new promising tool for the investigation of dislocation-related phenomena, phase transformations, grain boundary relaxations [44-48], and relaxation phenomena related to the movement of twin boundaries.

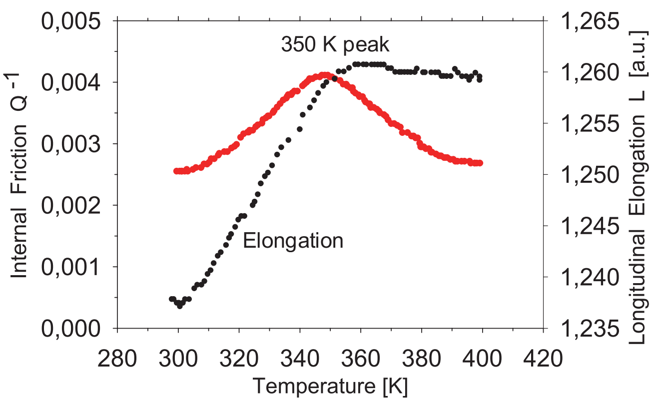


Fig. 4. Internal friction peak at 350 K in a Fe-C alloy after 4 % cold-work at 77 K, accompanied by longitudinal elongation of the sample measured by the laser triangulation technique (the resonant frequency is 1.4 Hz)

10. Conclusions

The high-resolution mechanical spectroscopy HRMS is established through recent advances in low-noise stress and strain signals, generation and control of high-quality stress and strain signals, and novel computing methods to estimate the internal friction and elastic modulus. It is demonstrated that the mechanical loss angle in forced oscillations is not determined by the precision in estimation of the phase angle between stress and strain signals. It is demonstrated that the resolution limit in the loss tangent is as low as $\tan\varphi_b = 1\times10^{-5}$. The highest estimation resolution of the dynamic modulus is around 1×10^{-5} in arbitrary units.

The importance of detrimental effects caused by zero-point drift observed during classical mechanical loss measurements (free decaying oscillations) is emphasized and two novel computing methods (YM and OMI), are advocated to eliminate the ZPD effect.

New perspectives for the development of mechanical spectroscopy coupled with an *in-situ* laser dilatometry are advocated to pave the way for a novel instrumentation in Materials Science.

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