Dynamic mechanical multi-harmonic analysis applied to elastomers characterization

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Elastomers exhibit very particular behaviour that can make it complex to characterize. The properties of such materials are sensitive to temperature, frequency, strain, and even sometimes to the dynamic and temperature history of the specimen itself. Measuring with high accuracy and high reproducibility dynamic properties of elastomers (E, G*, tangent delta) requires specific care. 01dB-METRAVIB has developed recently new and incomparable software called MULTITEST which extends capabilities of classical DMA.*

A new control principle makes possible to handle the strain really applied to the specimen with veryhigh accuracy, and to control various waveforms such as sine, pulse, triangle and user defined specific wave forms: it opens the door to test the elastomers as closed as possible of the final material application. Optimization of the dynamic excitation control (multi-harmonics) brings important information for non linear material characterization.

A specific function ELS (Excitation Level Storage) allows applying instantaneously the right amplitude and waveform to the specimen, after the material has been learned during a pre-test.

This new test-ware makes now possible to perform on classical DMA instrument, tests such as fatigue tests, heat built up tests and crack growth tests, additionally to all classical DMA capabilities.

This paper illustrates the benefits of DMA+ series combined with multi-harmonic analysis for the characterization of elastomers.

Key words: *dynamical mechanical analysis, testing instrument, elastomer properties*

Multiharmoniczna analiza dynamiczno--mechaniczna stosowana do określania właściwości elastomerów

Elastomery wykazują szczególne właściwości, przez co są one trudne do scharakteryzowania. Właściwości tych materiałów są zależne od temperatury, częstotliwości, odkształcenia, a nawet niekiedy od historii zmian dynamicznych i temperaturowych próbki. Uzyskanie dokładnych i wysoce powtarzalnych wartości pomiaru właściwości dynamicznych elastomerów (E, G*, tangens delta) wymaga szczególnej uwagi.*

Firma 01dB – METRAVIB opracowała ostatnio jedyne w swoim rodzaju oprogramowanie o nazwie MULTITEST, które rozszerza możliwości tradycyjnego DMA.

Nowa zasada sterowania pozwala z bardzo dużą dokładnością kontrolować naprężenie faktycznie występujące w próbce, a także stosować różne przebiegi drgań, np. sinusoidalne, pulsacyjne, trójkątne czy zdefiniowane przez użytkownika specyficzne postaci fali; otwiera to drzwi do badania elastomerów dokładnie w takich warunkach, w jakich znajdzie się końcowy wyrób. Optymalizacja (multiharmonicznego) sterowania pobudzeniem dynamicznym przynosi istotne informacje o nieliniowych właściwościach materiałów.

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Its most recent realizations are: ■ The development of an innovative METRAVIB instrument: VHF104, dedicated to direct high frequency materials mechanical testing up to 10kHz. ■ The development of a new patented 50 Newtons actuator for a tabletop METRAVIB Dynamic Mechanical Analyzer: DMA50. ■ Additionally to its involvement in the development of the material testing range of instruments Laurent PERIER is today Manager of the 01dB-METRAVIB testing laboratory.

Specyficzna funkcja ELS (Excitation Level Storage – zachowanie poziomu pobudzenia) pozwala poddać próbkę odkształceniom o właściwej amplitudzie i przebiegu natychmiast po zbadaniu materiału podczas testu wstępnego. Nowe oprogramowanie testowe umożliwia obecnie wykonanie na klasycznym urządzeniu DMA, poza standardowym zestawem badań, testów zmęczeniowych, generowania ciepła, wzrostu spękań. Artykuł ilustruje zalety wykorzystania urządzenia serii DMA+ połączonego z analizą multiharmoniczną do określania charakterystyki elastomerów.

Słowa kluczowe: *analiza dynamiczno-mechaniczna, urządzenie badawcze, właściwości elastomerów*

1. Introduction

Elastomers exhibit very particular behaviour. The properties of such materials are sensitive to temperature, frequency, strain, heat build up and even sometimes to the dynamic history of the specimen itself! Measuring with high accuracy and high reproducibility dynamic properties of elastomers (E*, G*, tangent delta) requires specific care.

The specimen preparation and its handling, the specifications and capabilities of the testing instrument, and the efficiency of the algorithm used to control the dynamic excitation are key points for the quality of the final measurement data. Measuring dynamic mechanical properties over a wide range of parameters (frequency, strain, temperature, excitation cycle,...) of materials is becoming more and more important, in order to understand and predict the behaviour of the material in its final configuration of excitation. Taking into account the dynamic behaviour of materials is becoming a key point to improve the design of products, and to ensure a perfect correspondence with the performances requirements.

2. DMA+ series

For more than 40 years, 01dB-METRAVIB is developing Dynamic Mechanical Analysis testing instruments. The technologies used to generate the excitation signal, to measure the displacement and force transferred to the specimen, have been developed to carry out high precision measurements of the complex modulus and tangent delta. The very high rigidity of the mechanical test frames, the powerful electrodynamic shakers are some of the key points of the DMA+ series instruments, that enables its high level of performances: force up to 450N, frequency range from static up to 1000 Hz, temperature from -150 ^{o}C up to 450 ^{o}C, wide range of accessible strain (up to 300%).

Figure 1. *METRAVIB DMA + 150*
Rys. 1. *Urządzenie METRAVIB DMA+150*

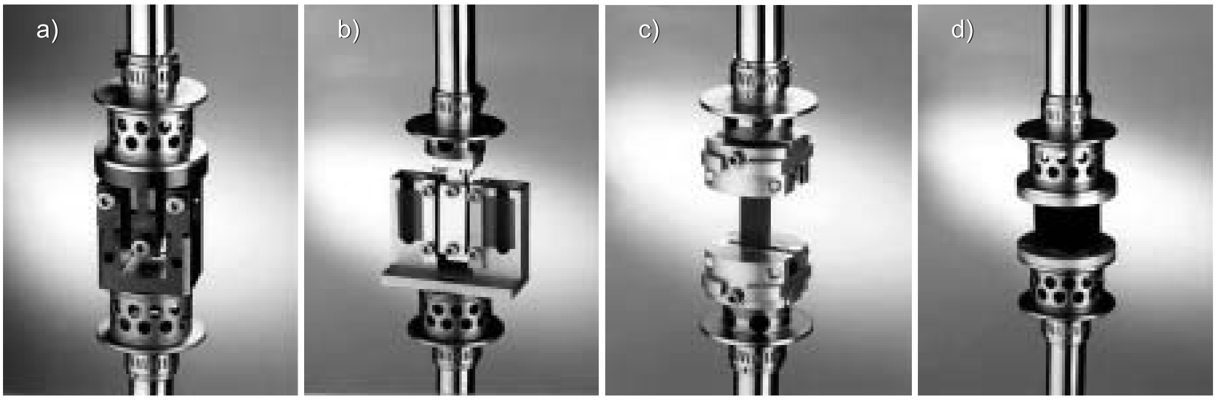

Figure 2. *Specimen holders currently used for elastomers testing: a) shear for sandwiches, b) shear for films, c) tension jaws, d) compression plates*
Rys. 2. *Uchwyty próbek używane obecnie w badaniach elastomerów: a) ścinające do badania próbek wielowarstwowych, b) ścinające do filmów, c) szczęki rozciągające, d) płytki ściskające*

Each METRAVIB DMA+ (figure 1), can use a large range of specimen holders allowing different testing modes. Tension, compression, shear and bending tests are possible on the same instrument. The optimization of the dimensioning of the specimen can be easily made by using a software dedicated wizard; the same wizard helps the operator in choosing the adequate specimen holder according to the purpose of the test. Some tests require the use of glue to fix the specimen on the specimen holder; one of the particularities of elastomers is that these materials are not always compatible with glues. Furthermore the typical temperature range that is addressed during the test makes also bring additional constraints to find the appropriate glue suitable for both elastomer and the required range of temperature.

Typical temperature range can goes from very low temperature (-150^{o}C) up to possibly high temperature.

Figures 2 below is presenting specimen holders frequently used for elastomers testing. They allow fixing the material's specimen by efficient clamping of the specimen in the whole temperature range of interest.

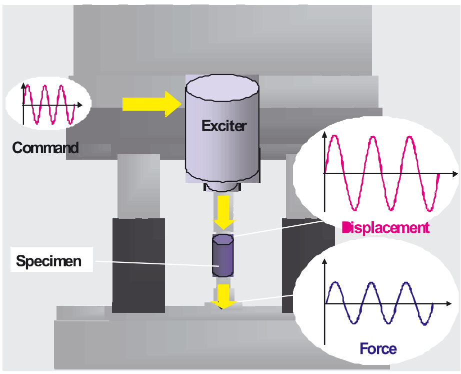

Figure 4. *Classical DMA principle*
Rys. 4. *Zasada działania klasycznego DMA*

3. Reminder of classical dynamic mechanical analysis (DMA or DMTA)

Conventional dynamic mechanical thermal analysis (DMTA) makes possible to study the behaviour of elastomers versus temperature, strain or stress, frequency (cf. figure 3).

Storage shear modulus G' and tan delta vs. temperature, strain and frequency

a) Temperature ramp from -150^{o}C up to 30^{o}C.

b) Strain sweep from 0.1% to 300%

c) Frequency sweep from 1 Hz to 1000 Hz.

As shown in Figure 4, this principle of classical DMA consists in applying, using an electrodynamic actuator, a sine excitation to the specimen, out of resonance mode.

Then, knowing the specimen dimensions, one can calculate the intrinsic characteristics of the material (complex modulus, loss angle) from the measured force and displacement.

The conditions under which a DMA test is performed allow to impose a displacement or a force, at a given frequency or amplitude. Several iterations are required before the amplitude set point could be reached. During each iteration, the value of the magnitude under monitoring (force or displacement) is compared to the set point. The value of the sine command is then changed and a new measurement is carried out. The measurement is performed when the current amplitude has reached the set point (cf. figure 5).

For non-linear materials, force and displacement signals are not pure sine signals. However, according to standards, a DMA instrument uses only the first harmonic to compute the various magnitudes. It leads to the limitations of conventional DMA:

- The sinusoidal solicitation mode can be not representative to the real-life conditions of use for the final product.

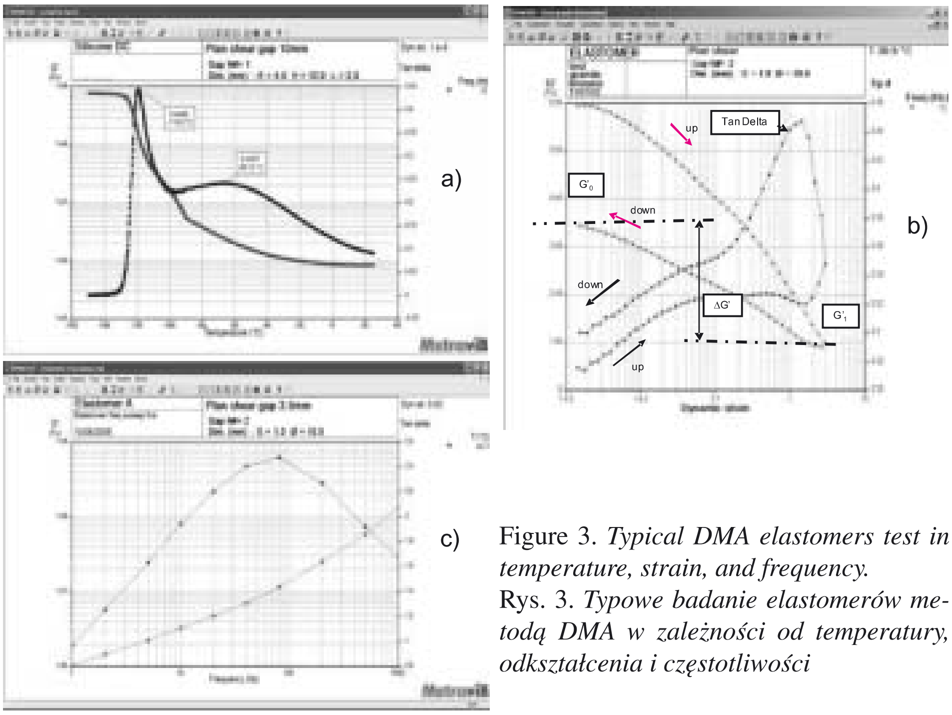

Figure 3. *Typical DMA elastomers test in temperature, strain, and frequency.*
Rys. 3. *Typowe badanie elastomerów metodą DMA w zależności od temperatury, odkształcenia i częstotliwości*

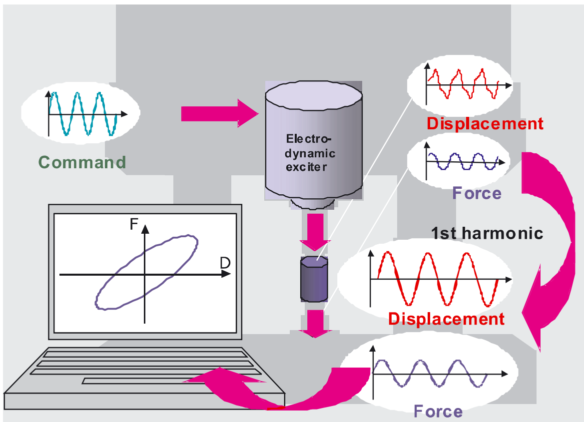

Figure 5. *Typical control loops in a classical DMA test*
Rys. 5. *Typowe pętle sterowania w klasycznym teście DMA*

- DMA can theoretically be applied to linear materials behaviour; in fact nowadays its application has become common as well for non-linear materials.

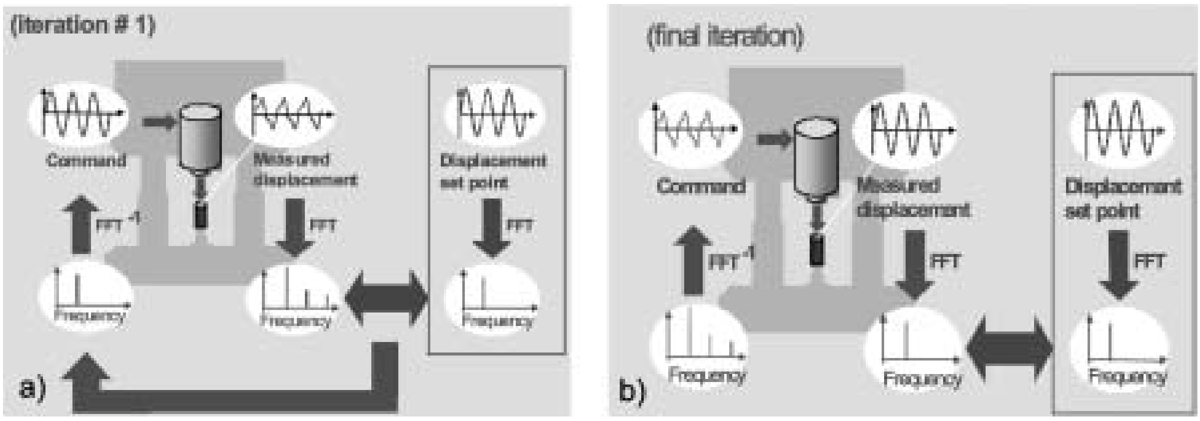

Figure 6. *MULTITEST – Multi harmonic dynamic mechanical analysis – a) first and b) last iteration of the control loop*
Rys. 6. *MULTITEST – Multiharmoniczna analiza dynamiczno-mechaniczna – a) pierwsza i b) ostatnia iteracja sterowania cyklem*

- DMA does not control the waveform that is actually applied to the material's specimen.
- The number of cycles imposed to the specimen in order to reach the set point can change the characteristics that are measured.

4. Multitest: using of multi-harmonic excitation in dynamic mechanical analysis for the characterization of elastomers

The control principle is based on the comparison of the measured waveform to the target waveform. To do so, MULTITEST software compares the FFT of the measured displacement signal to the FFT of the displacement set point. The command signal is modified according to the rneasured deviations (cf. figure 6a). After a few iterations (cf. figure 6b), the command signal is no longer sinusoidal, but the deformation applied to the specimen is compliant with the defined setting. This new approach makes possible to control both the amplitude and the waveform of the distortion actually applied to the specimen.

5. Characterization elastomers behavior versus the wave form applied

The multi-harmonic control principle makes possible to handle the deformation really applied to the specimen with very high accuracy, but also to control various waveforms such as sine, pulse, triangle and user defined specific ones: it makes then possible elastomers testing under conditions as close as possible of the final material application.

Sine wave

Strain sweep, frequency sweep are possible from very low strain up to high stain. The loops stress / versus strain are recorded from the first cycle up to the last one (cf. figure 7).

Generally the lost factor of the material for a sine excitation is computed from the complex modulus ratio E"/E' in tension compression or G"/G' in shear. Thanks to MULTITEST, the loss factor Tan delta can also be determined from dissipated (EpsD) and provided (EpsF) energies during each cycle (cf. figure 8).

When materials are sensitive to Mullins / Payne effect the Force – displacement loops tends to turn toward the X axis versus number of cycles (cf. figure 9).

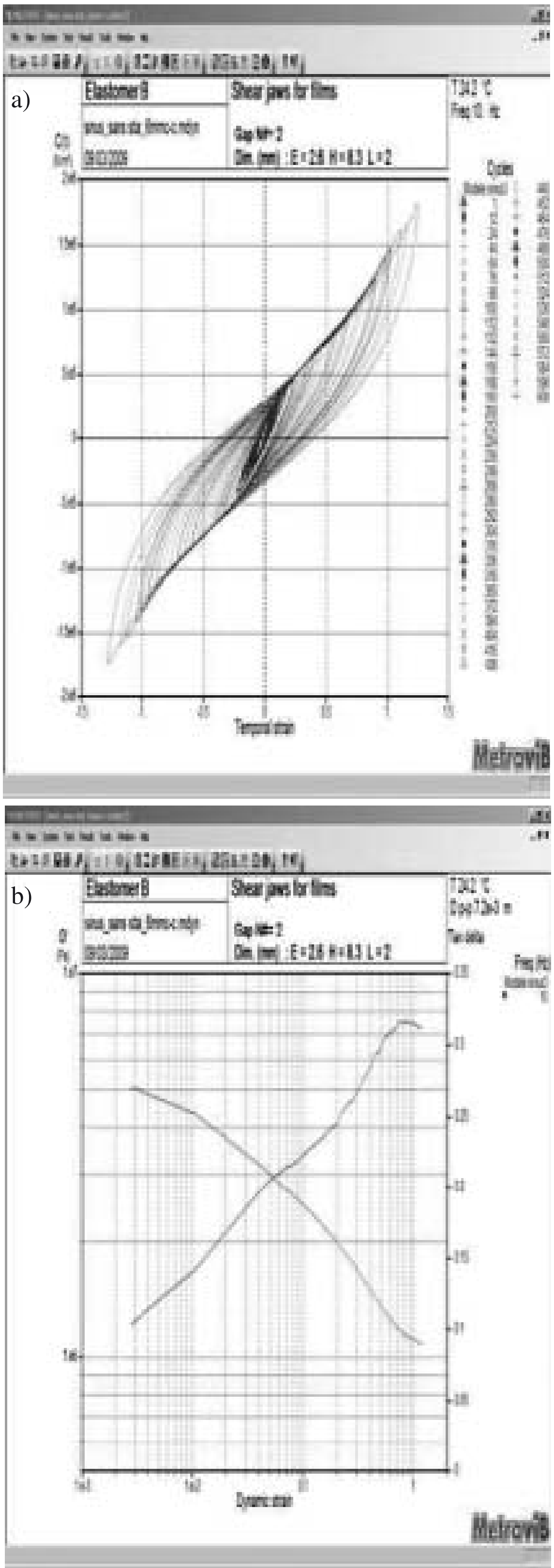

Figure 7. *Elastomer – shear test – strain sweep up to 125% – a) Stress/strain loops at various cycles number b) storage shear modulus G' and Tan delta vs. dynamic strain (0-peak).*
Rys. 7. *Badanie ścinania – Zmiana odkształcenia do 125% – a) Pętle histerezy naprężenie/odkształcenie przy różnej liczbie cykli; b) Moduł zachowawczy G' i tangens delta w funkcji odkształcenia dynamicznego (0-pik)*

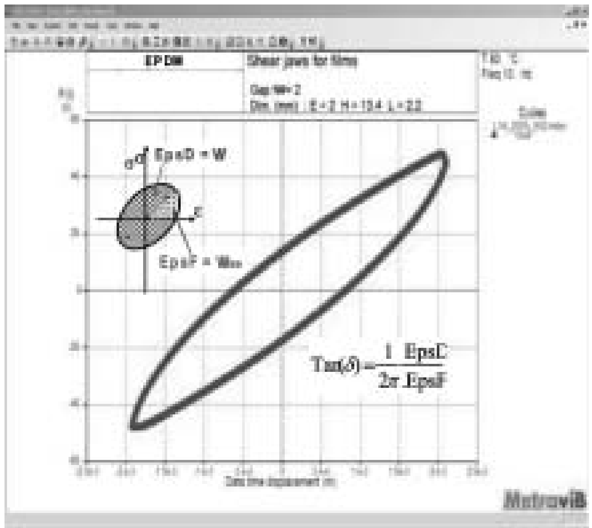

Figure 8. *Loss factor Tan delta determined from one hysteresis loop.*
Rys. 8. *Współczynnik stratności tangens delta określony na podstawie jednej pętli histerezy*

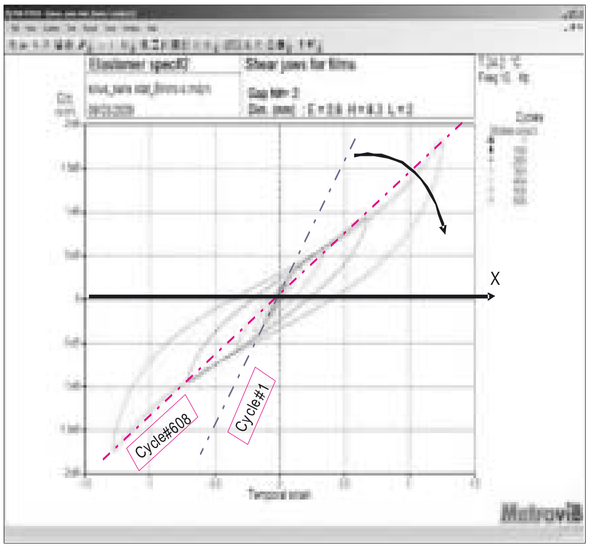

Figure 9. *Elastomer – Mullins / Payne effect*
Rys. 9. *Elastomer – Efekt Mullinsa/Payne'a*

Specific wave forms

Sometime, the sine solicitation mode can be not exactly representative to the real-life conditions of use for the final product. For instance, let's consider a part of a tire tread. The load is applied when this part is in contact with the road surface. During the rest of the rotation time, the load is no longer applied.

In the example bellow, a DMA+ instrument was used to reproduce the dynamic excitation corresponding to the final use conditions of an industrial product. MULTITEST software makes possible to analyze the material's response to excitation wave form other than sinusoidal: the applied excitation wave form is customized. This is possible in importing an Excel file including the waveform to apply. In this example, the

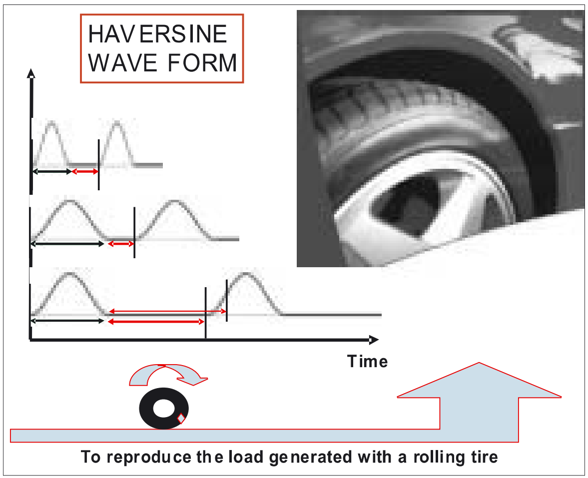

Figure 10. *Haversine wave form – capabilities to simulate rolling behaviour*
Rys. 10. *Odkształcenia półsinusoidalne – symulacja zachowania się materiału podczas toczenia*

customized waveform is the addition of two sinusoidal signals of different frequencies freq1 and freq2:

- Frequency ratiofreq2/freq1 = 30.
- Relative amplitudes: 100% at freq1 and 10% at freq2.

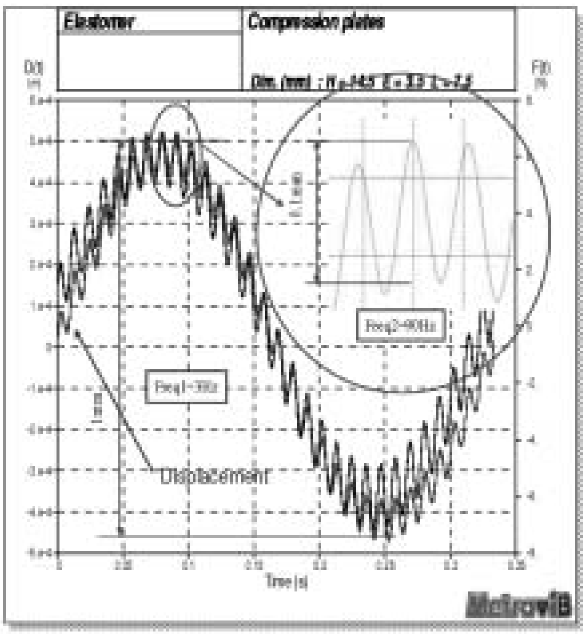

Figure 11. *Customized waveform to fit with the material final use conditions – 1 mm sine displacement at 3 Hz superposed with 0.1mm sine displacement at 90Hz*
Rys. 11. *Przebieg drgań dostosowany do warunków użytkowania gotowego wyrobu – odkształcenia sinusoidalne o amplitudzie 1 mm i częstotliwości 3 Hz nałożone na drgania o amplitudzie 0,1 mm i częstotliwości 90 Hz*

6. Testing elastomers in fatigue

Control of the excitation

When elastomer compounds present very non linear behaviours, it is important to get on one hand instantaneously the right setting values, i.e. strain or stress, and on the other hand the first hysteresis loop of stress versus deformation.

It is why the first cycle of deformation is always recorded by MULTITEST, as well as the ELS (Excitation Level Storage) specific function allows applying instantaneously the right setting amplitude and waveform to the specimen from the first cycle, after the material has been learned during a pre-test with a first specimen.

Heat build up

If no specific frequency value is required for the test, DMA+ machine makes possible to optimize the test duration by increasing the test frequency. However, when imposing high strain or stress and high frequency to an elastomer specimen, the applied energy is transformed into heat inside the specimen: this phenomenon is known as heat build up.

The heat build up depends on strain ε, frequency *f*, loss factor tan (δ), storage Young modulus *E'* and specimen volume V_s (cf [1]).

$$P_d = 2\pi f \cdot \tan(\delta) E' \varepsilon^2 V_s \quad (1)$$

If the frequency is increased in order to optimize the test duration, it is necessary to verify that the heat build up phenomenon is negligible. Actually, the model DMA+450, is especially well adapted to study this phenomenon, in making possible applying high strain excitation. An additional thermal probe may be used to accurately measure the specimen temperature changes. This additional probe can be located at different places depending on testing requirement (standard): close to specimen surface, inside specimen, inside specimen's supports (cf. figure 12).

Fatigue test and extended application of results

The fatigue tests are generally performed in order to estimate the life time or the evolution of the behaviour of several blends of compounds. Figure 13 corresponds to a test performed in compression with a strain of 25% peak-to-peak with three elastomer specimens referenced A, B and C. A and C material are relatively strong to the fatigue (ΔK/K ≈ 8% after 2E6 and 3E6 cycles).

The behavior of the specimen C versus fatigue differs from the two other specimens: the stiffness relative variation after 2E6 cycles is significant (ΔK/K ≈ 40%).

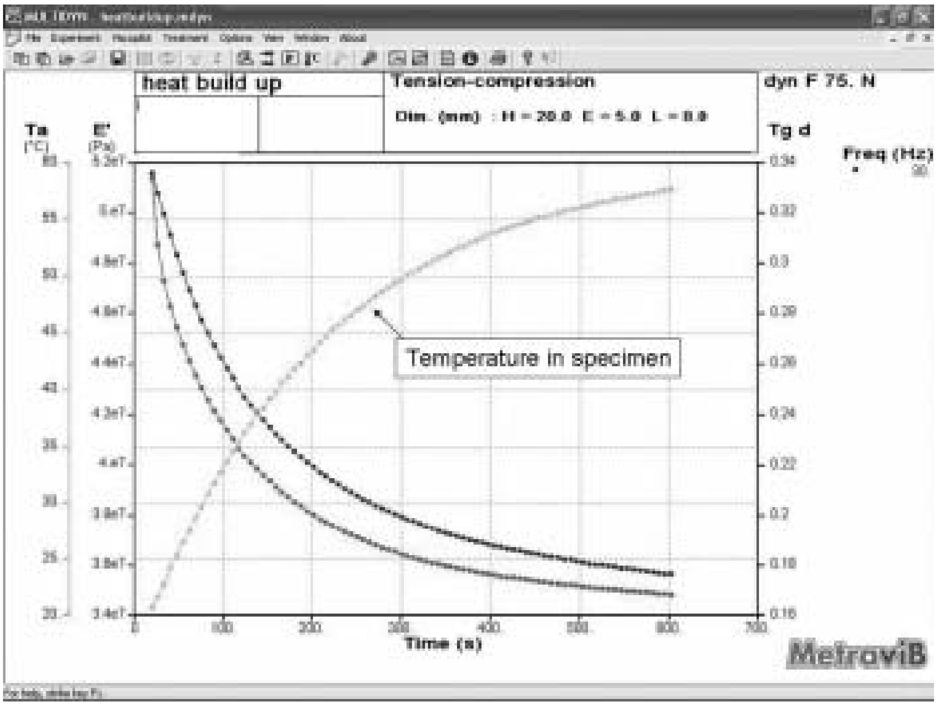

Figure 12. *Elastomer – Heat build up phenomenon.*
Rys.12. *Elastomer – Zjawisko generowania ciepła*

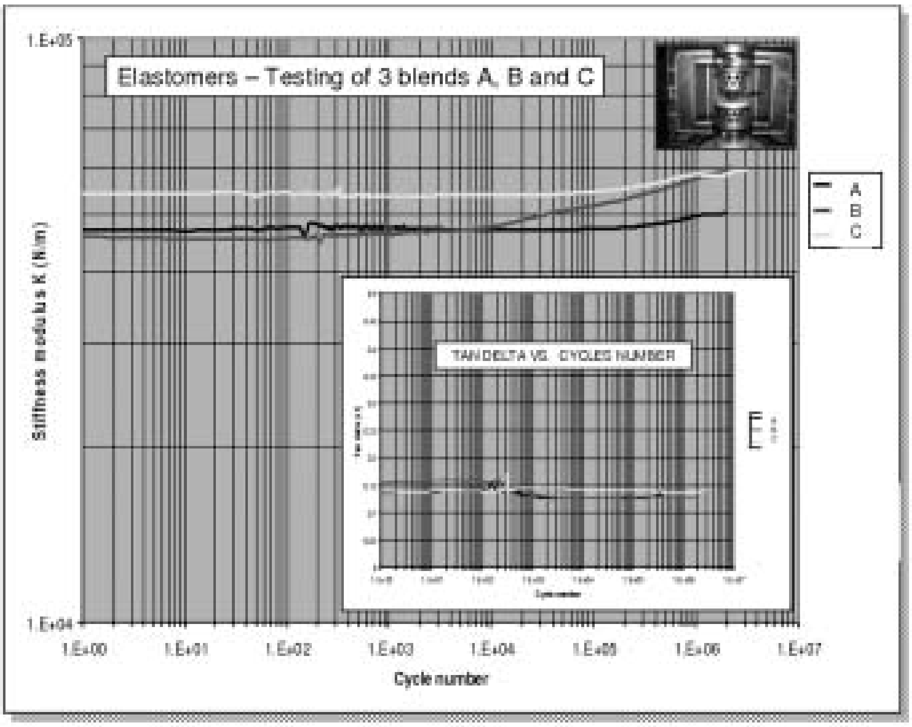

Figure 13. *Testing in fatigue of 3 elastmer blends used for the same type of final application.*
Rys. 13. *Badanie zmęczeniowe 3 mieszanek elastomerowych o tym samym przeznaczeniu.*

Complementary tests can be performed with MULTITEST in tension or shear mode, in order to evaluate the materials behaviour in a wide range of dynamic strain (up to 300% in shear), at various steps of strain. The end of the fatigue test depends on the damaged criteria for the study: it can be the breaking of the specimen or a given variation of the specimen's stiffness (i.e. 50%). Figure 14 presents a typical result obtained at one strain value.

Then the equivalent Basquin curves for elastomers can then be post processed and used in fem modeling.

Basquin's ratio b and C are described by [2]. The results obtained during the fatigue test at various steps of strain are presented according to [3J.

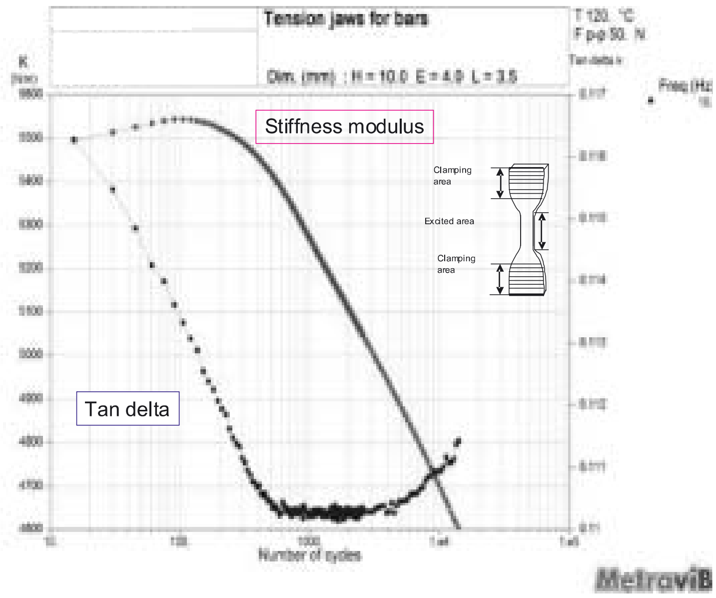

Figure 14. *Elastomer – Fatigue test with MULTITEST up to the breaking of the specinen at a given strain.*
Rys. 14. *Elastomer – Test zmęczeniowy sterowany MULTITESTEM prowadzony do zniszczenia próbki przy danym odkształceniu*

Figure 16. *DMA+300 equipped with the crack growth test module.*
Rys. 16. *Urządzenie DMA+300 wyposażone w moduł badania wzrostu spękań*

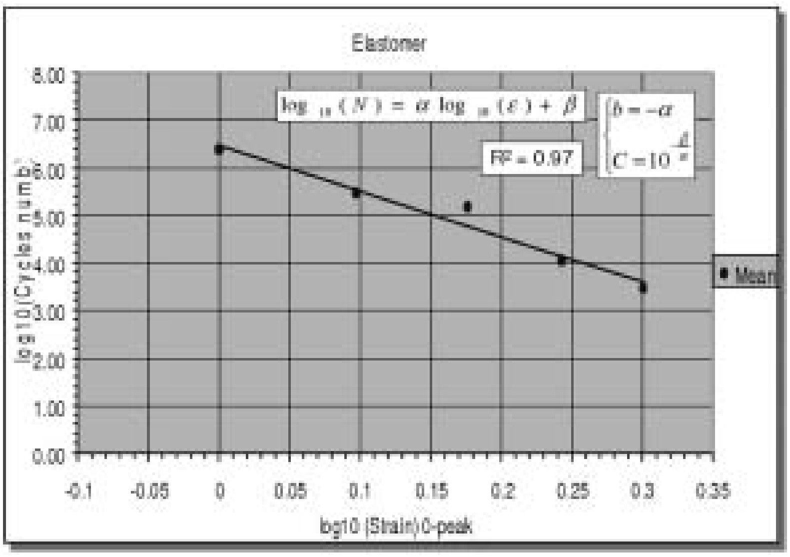

Figure 15. *Post processing of the equivalent of Basquin's ratio for the elastomer tested.*
Rys. 15. *Dalsza obróbka danych: równoważny współczynnik Basquina dla badanego elastomeru*

$$N\varepsilon^b = C \quad (2)$$
$$\log_{10}(N) + b \log_{10} \varepsilon = C \quad (3)$$

These Basquin's ratios are generally used in order to predict the life time of product at lower strain than tested. This corresponds to a very practical way to interpolate breaking data for strain values, that have not been analyzed, and then to save testing time.

7. Crack growth tests

A better understanding of crack growth behaviour in elastomer or the study of the material resistance against crack growth can be another important point in product knowledge. DMA+300 is particularly well adapted to this type of test when equipped with the crack growth test module which incorporates cutting and optical systems (cf. figure 16).

The operator initiates cracks in the specimen on left and right sides by using a dedicated tool of the machine. The initial crack length is about few millimetres in order to initiate the cracking The crack growth test consists in following the crack growth according to solicitation and environmental test conditions. Figure 17 corresponds to the crack growth speed on twice side of the elastomer specimen.

8. Conclusion

Elastomers are complex materials that require accurate testing.

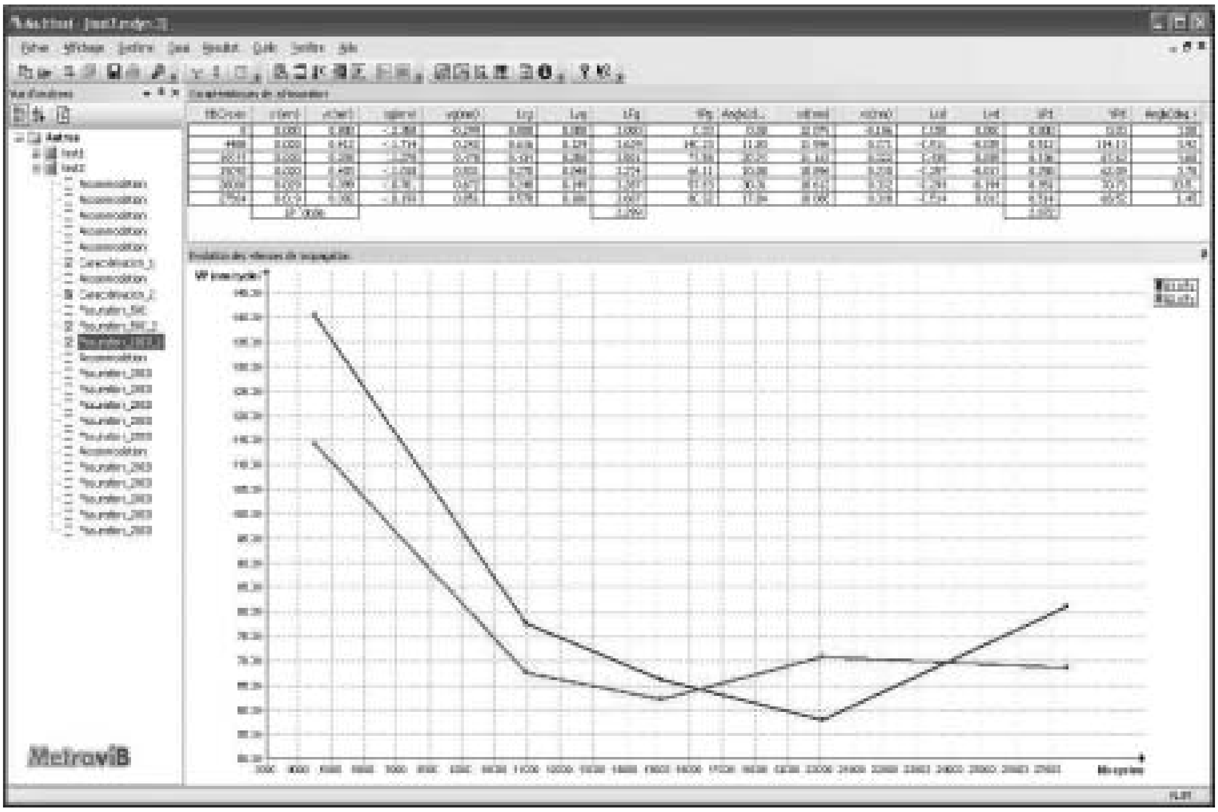

Figure 17. *Elastomer – Crack growth measurement – Crack growth speed in nm/cycles vs. cycles number.*
Rys. 17. *Elastomer – Pomiar wzrostu spękań w nm/cykl w zależności od liczby cykli*

The good knowledge of the performances of the material, are essential compounders, researchers and modeling engineers.

The data obtained with DMA+ series can be used as well in research domain to understand, characterize and compare new compounds, as in quality control domain to ensure that the production process is producing material with expected properties.

The METMVIB DMA+ series is offering a very unique range of tests capabilities and testing performances.

Thanks to their wide range of specifications (force up to 450 Newtons, frequency from static up to 1000 Hz, temperature from –150°C to 450°C), DMA+ series enables to characterize accurately dynamic and static properties of material, as for example: the temperature, strain (Payne and Mullins effect) and frequency effects. With the full range of specimen holders available, it is easy to accommodate the instrument with different geometries of specimen (sheet, cylinder, fibres), as well as with the different natures of material (liquid, paste, gel, powder and solid).

Thanks to new MULTITEST software, DMA+ series allows to perform on one single instrument, fatigue testing, crack growth tests in order to understand the material's mechanical properties evolution under different excitation conditions and different excitation waveforms.

The possible application on the instrument of excitation corresponding to vibrations encountered by the manufactured component allows providing more accurate data on the lifetime of the product.

When controlled accurately, increasing the frequency of excitation enables to get fatigue information faster, and so to save significantly testing time. Further to the different tests capabilities presented in this document, DMA+ series also proposes additional capabilities such as hygrometry control chambers, crack growth follow up system.