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Validation of the IMCE RFDA System for Dynamic Elastic Moduli Measurements



Anne A. Campbell
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March, 2015

OAK RIDGE NATIONAL LABORATORY

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Materials Science & Technology Division

**VALIDATION OF THE IMCE RFDA SYSTEM FOR DYNAMIC ELASTIC
MODULI MEASUREMENTS**

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ACRONYMS

LAMDA	Low Activation Materials Development and Analysis Laboratory
IMCE	Integrated Material Control Engineering
RFDA	Resonant Frequency and Damping Analyzer
DAC	Data Acquisition Card
GUI	Graphical User Interface
FFT	Fast Fourier Transform

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ABSTRACT

The focus of this report is to describe the new system that has been acquired for the Low Activation Material Development and Analysis Laboratory (LAMDA) to perform the measurement of the dynamic Young's and shear moduli, and to show that the results of the new system are more easily analyzed and more accurate than the current systems. The current system is the GrindoSonic MK5 Impulse Excitation and Vibration Test System from J.W. Lemmens, Inc., and the new system is the Resonant Frequency and Damping Analyser (RFDA) from Integrated Material Control Engineering (IMCE). Overall both systems were adequate for measuring the Young's modulus, but the ease of setup and consistency of strike repetition makes the RFDA system the better option. Care must be taken when measuring shear modulus with the GrindoSonic, because it is necessary to calculate the approximate frequency of the shear peak to ensure the correct peak is measured. A comparison between the results from both systems was performed with nine specimen shapes and five graphite grades. 15 of the 17 measured grade/shape comparisons showed excellent consistency of the measured moduli from both systems, values agreed within 1-2%. The only outliers were a 20% disagreement for one specimen shape and a 5-10% disagreement for a second specimen shape for shear modulus, but it was shown that this occurred because the GrindoSonic system was reporting the frequency for the flexural harmonic peak rather than the fundamental torsional peak.

1. MEASUREMENT OF DYNAMIC YOUNG'S AND SHEAR MODULI

The measurement of the dynamic elastic moduli for ceramics and graphite are discussed in ASTM standards C747-93 [1], C1198-09 [2], and C1259-14 [3]. These three standards have two distinct methods of activating the dynamic vibration of the specimens. C747-93 and C1198-09 both use a variable oscillation driver to continuously induce the vibration modes, while C1259-14 uses a single elastic physical strike to induce the vibrations. The vibration of the specimen (via either method) is measured with a transducer, either contact (accelerometer) or non-contact (microphone), which converts the mechanical vibrations into electrical signals. The electrical signal is analyzed to determine the fundamental frequency, or resonance period. The analysis of the vibrations (according to C747-93 and C1198-09) is performed with an oscilloscope that has the driver oscillation frequency as the independent variable and the transducer recorded vibration of the specimen as the dependent variable. The fundamental frequency is then determined by sweeping the oscillation driver over a range of frequencies until the frequency that produces the greatest specimen displacement is found. The striking method instead records the vibration of the specimen as it undergoes relaxation, and then a Fourier analysis is used to determine the resonance frequencies.

2. SPECIMEN SETUP FOR MODULUS MEASUREMENT

Performing the dynamic modulus measurements requires dampening vibrations at specific locations on the specimens (nodes). The setup used for the Young's modulus (flexural vibration) measurement requires striking the center of the specimen with the transducer located at one of the ends of the specimen, and utilizes a two-node setup where the nodes are perpendicular to the length and located 0.224 times the length in from the ends of the specimen (Fig. 1). The ratio of the first four harmonics to the fundamental flexural frequency are not integers, rather the ratios are:

$$1 : 2.757 : 5.404 : 8.933 : 13.344 \dots [4].$$

These ratios are for specimens with a very low thickness to length ratio, but as the thickness to length ratio increases then these harmonic ratios decrease. The setup for the shear modulus (torsional vibration) measurement involved striking the specimen at one corner, with the transducer located at the corner

diagonally opposite the strike point, and utilizes two nodes: one that is parallel to the specimen length and located along the centerline of the width and one that is parallel to the specimen width and located along the centerline of the length (Fig. 2). The ratios of the torsional harmonics are linear with the harmonic number (i.e. the second harmonic frequency is two time larger than the fundamental frequency).

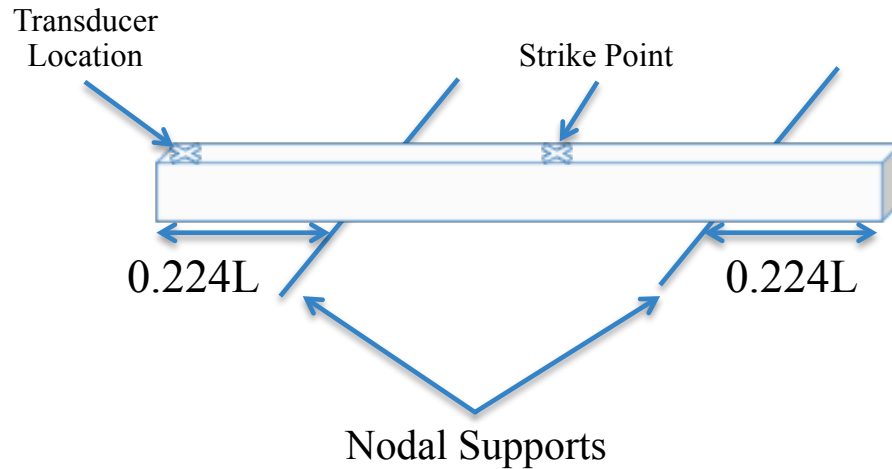


Fig. 1. Schematic drawing of the flexural setup for measuring Young's modulus of beam-shaped specimens.

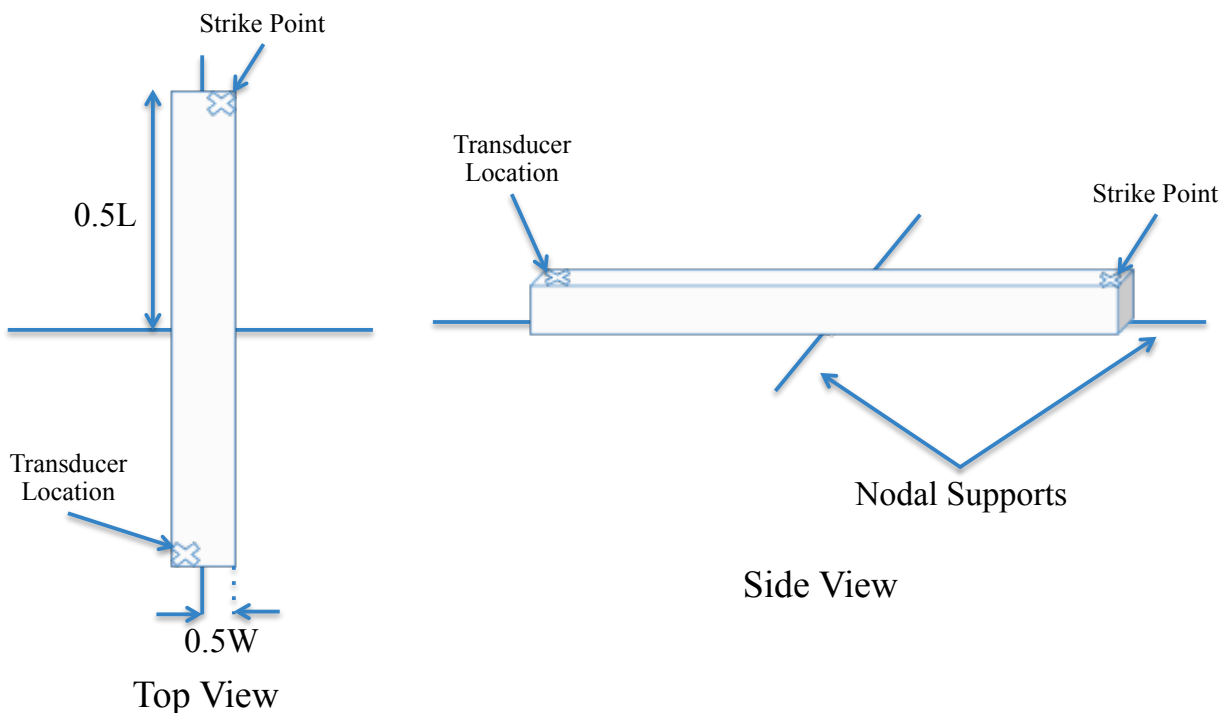


Fig. 2. Schematic drawing of the torsional setup for measuring shear modulus of beam-shaped specimens.

3. GRINDOSONIC MK5 SYSTEM

The first system at Oak Ridge National Laboratory (ORNL), for measuring the elastic properties, is the GrindoSonic MK5 Impulse Excitation and Vibration Test System from J.W. Lemmens, Inc. This system is a very simple setup, using a metal ball bearing attached to the end of a wire zip-tie as the impulser, a microphone transducer, and an electrical module that performs all the signal analysis (Fig. 3).



Fig. 3. Photographs of the GrindoSonic MK5 system. The left photograph shows the impulser (wire zip-tie with ball bearing attached to end), and the microphone transducer. The right photograph shows the enclosed electronic module, which contains the signal amplifier, and frequency analyzer, and displays the fundamental frequency calculated by the electronics.

The one inherent problem with this system is that the frequency displayed by the electronic module corresponds to the “purest waveform”, according to the GrindoSonic manual [5], from an unknown analysis method of the measured peaks. This description of how the displayed frequency is determined is vague, but it is thought that the displayed frequency is for the vibrational peak with the highest amplitude. This method of determining the fundamental frequency is suitable for flexural measurements (Young’s modulus), but can possibly produce errors when measuring the torsional frequency (shear modulus). The cause of this possible error with the torsional setup is that there is no way to setup the specimen so as to only activate torsional vibration without also causing some flexural vibrations. Therefore it is possible that during the torsional measurements, the reported frequency maybe actually be one of the resonances of the flexural vibration, rather than the desired torsional vibration.

4. COMPARISON OF ORNL AND MANUFACTURER VALUES OF YOUNG’S MODULUS

Four graphite grades, with nine different specimen shapes/sizes, were used to verify that the new system for measuring the Young’s and shear moduli. Before comparing systems, it is first imperative to compare the measurements with the original system to the values reported by the manufacturers. The Young’s modulus values quoted by the respective manufacturers are listed in Table 1. A summary of the ORNL measured Young’s and shear moduli, measured with the GrindoSonic system, is listed in Table 2, where the values are the average and one standard deviation. From these two sets of values, it is apparent that there is a directional dependence of both moduli, with a greater effect on the Young’s values. The orientation of the measurements from the manufacturers is unknown.

Table 1. Young's modulus values reported by graphite manufacturers

Manufacture	Graphite grade	Young's modulus (GPa)
Ibiden	ETU-10	9.7 - 9.8 ^a
Tokai	G347A	11.5 ^b
Toyo Tanso	IG-110	9.8 ^a
Toyo Tanso	IG-430	10.8 ^a
NTC	IGS-734H	not available

^a Modulus measurement method unknown^b Modulus measured via sonic velocity**Table 2. Young's and shear modulus values, measured at ORNL with the GrindoSonic system, for various specimen dimensions and orientations.**

Specimen dimensions (mm)	Specimen shape ID	Manufacture	Graphite grade	Number of specimens	Young's modulus (GPa)	Shear Modulus (GPa)
30 x 3 x 2.6	SB3	Ibiden	ETU-10	48 AX	9.23 ± 0.12	
				48 TR	10.44 ± 0.16	
24 x 5 x 1	MB5	Ibiden	ETU-10	61 AX	8.67 ± 0.13	3.40 ± 0.07
				60 TR	10.14 ± 0.24	3.96 ± 0.07
50 x 4 x 2	YMFS	Ibiden	ETU-10	10 AX	9.65 ± 0.15	
				10 TR	10.71 ± 0.09	
75 x 15 x 3	SMFS	Ibiden	ETU-10	10 AX	9.70 ± 0.12	4.59 ± 0.03
				10 TR	10.35 ± 0.08	4.80 ± 0.02
30 x 3.8 x 2.9	SB-W4	Tokai	G347A	64 AX	11.14 ± 0.15	
				60 TR	11.3 ± 0.34	
48 x 6 x 1	MB-W6	Tokai	G347A	60 AX	10.33 ± 0.16	4.15 ± 0.06
				10 TR	10.70 ± 0.05	4.23 ± 0.03
50 x 4 x 2	E-FS	Tokai	G347A	10 AX	10.46 ± 0.04	
				10 TR	10.88 ± 0.03	
70 x 15 x 3	MB-FS	Tokai	G347A	10 AX	10.96 ± 0.24	4.10 ± 0.08
				9 TR	11.10 ± 0.60	4.12 ± 0.01
25 x 3 x 2.9	MB	Toyo Tanso	IG-110	64 AX	9.10 ± 0.22	
				64 TR	9.69 ± 0.22	
25 x 3 x 2.9	MB	Toyo Tanso	IG-430	24 AX	10.61 ± 0.16	
				33 TR	11.24 ± 0.39	
24 x 3.5 x 2.25	CB	NTC	IGS-743H	62 AX	9.79 ± 0.08	
				62 TR	10.70 ± 0.09	
48 x 6 x 1	MB	NTC	IGS-743H	24 AX	9.58 ± 0.08	4.03 ± 0.08
				33 TR	10.59 ± 0.09	4.72 ± 0.31

The difference between the manufacturer quoted Young's modulus, and the ORNL measured directionally dependent Young's modulus is given in Table 3. The values listed for comparison are the percent difference between the manufacturer values and the directionally dependent values, and the average of the two directional values. These differences between the manufacturer quoted values, and the ORNL measured values may seem large but this is most likely an artifact of the measurement process and

the manufactures reporting a single value rather than ones that are also directionally dependent. Not knowing the method used by the manufacturer adds another level of discrepancy between the two values. Recent work from Burchell [6] found that Young's modulus measurements from dynamic measurements were up to 15% higher than the same value from sonic velocity measurements, for the same specimens. Whereas, work from Carroll [7] found dynamic Young's modulus was up to 7% smaller than sonic velocity modulus. Both of these discrepancies agree with comments in ASTM C-769 [8], where the accuracy of the sonic velocity measurements were noted to only be within 10% accuracy. Therefore, the difference between the ORNL measured values, and the manufacturer quoted values (ORNL values being anywhere from 7% larger to 11% smaller), are within a range of agreement that has been observed for other graphite measured with different techniques.

Table 3. Summary of the agreement between the ORNL measured Young's modulus and the values quoted by the manufacturers.

Specimen dimensions (mm)	Specimen shape ID	Manufacture	Graphite grade	Orientation	$(E_{\text{Manufacturer}} - E_{\text{ORNL}})/E_{\text{Manufacturer}} (\%)$	
					Directionally dependent	Direction average
30 x 3 x 2.6	SB3	Ibiden	ETU-10	AX	5.33	-0.56
				TR	-7.08	
24 x 5 x 1	MB5	Ibiden	ETU-10	AX	11.08	3.83
				TR	-4.00	
50 x 4 x 2	YMFS	Ibiden	ETU-10	AX	1.03	-4.09
				TR	-9.85	
75 x 15 x 3	SMFS	Ibiden	ETU-10	AX	0.51	-2.51
				TR	-6.26	
30 x 3.8 x 2.9	SB-W4	Tokai	G347A	AX	3.13	2.43
				TR	1.74	
48 x 6 x 1	MB-W6	Tokai	G347A	AX	10.17	8.57
				TR	6.96	
50 x 4 x 2	E-FS	Tokai	G347A	AX	9.06	7.22
				TR	5.39	
70 x 15 x 3	MB-FS	Tokai	G347A	AX	4.70	4.09
				TR	3.48	
25 x 3 x 2.9	MB	Toyo Tanso	IG-110	AX	7.14	4.13
				TR	1.12	
25 x 3 x 2.9	MB	Toyo Tanso	IG-430	AX	1.76	-1.16
				TR	-4.07	

5. IMCE RFDA SYSTEM

The new dynamic modulus system at ORNL is the Resonant Frequency and Damping Analyser (RFDA) from Integrated Material Control Engineering (IMCE). A picture of the setup is shown in Fig. 4. This system includes a wire specimen support that has movable wires to accommodate a range of specimen sizes, an automatic impulser (or can be used with the ball bearing on a wire zip-tie), a microphone transducer to measure sample vibration, the digital acquisition cards (DAC), and a computer with RFDA software to record, analyze, and save the results of each measurement. This system provides the user more control over the measurement and analysis process, especially for specimens where the torsional setup causes the flexural vibrations to be as high, or be higher than, the amplitude of the torsional vibrations.



Fig. 4. Photograph of the IMCE RFDA professional setup (picture from <http://www.imce.eu/rfda-mf-professional>).

The user has complete access and control of the analysis process while performing the modulus measurements, through the graphical user interface (GUI) shown in Fig. 5. This GUI allows the user to view the signal recorded by the microphone transducer (top left graph), control the parameters of and view the results of the Fast Fourier Transform (FFT) of the recorded signal (top right graph), and also displays information regarding the specimen identification and analysis procedure. In addition to processing the data during acquisition, the user can also post-process the saved data, with a screen shot of this GUI being shown in Fig. 6 and Fig. 7, for Young's and shear measurements, on the same specimen, respectively. Lastly, the user can plot multiple spectra on one plot to easily show different behaviors, an example from this option is shown in Fig. 8 using the same spectra from Fig. 6 and Fig. 7. The plotting of both spectra in Fig. 8 shows that flexural vibrations are still activated during torsional measurement, hence why the four peaks in the flexural spectrum (red) are also present in the torsional spectrum (blue). These user interfaces allow better determination of the different peaks for each analysis.

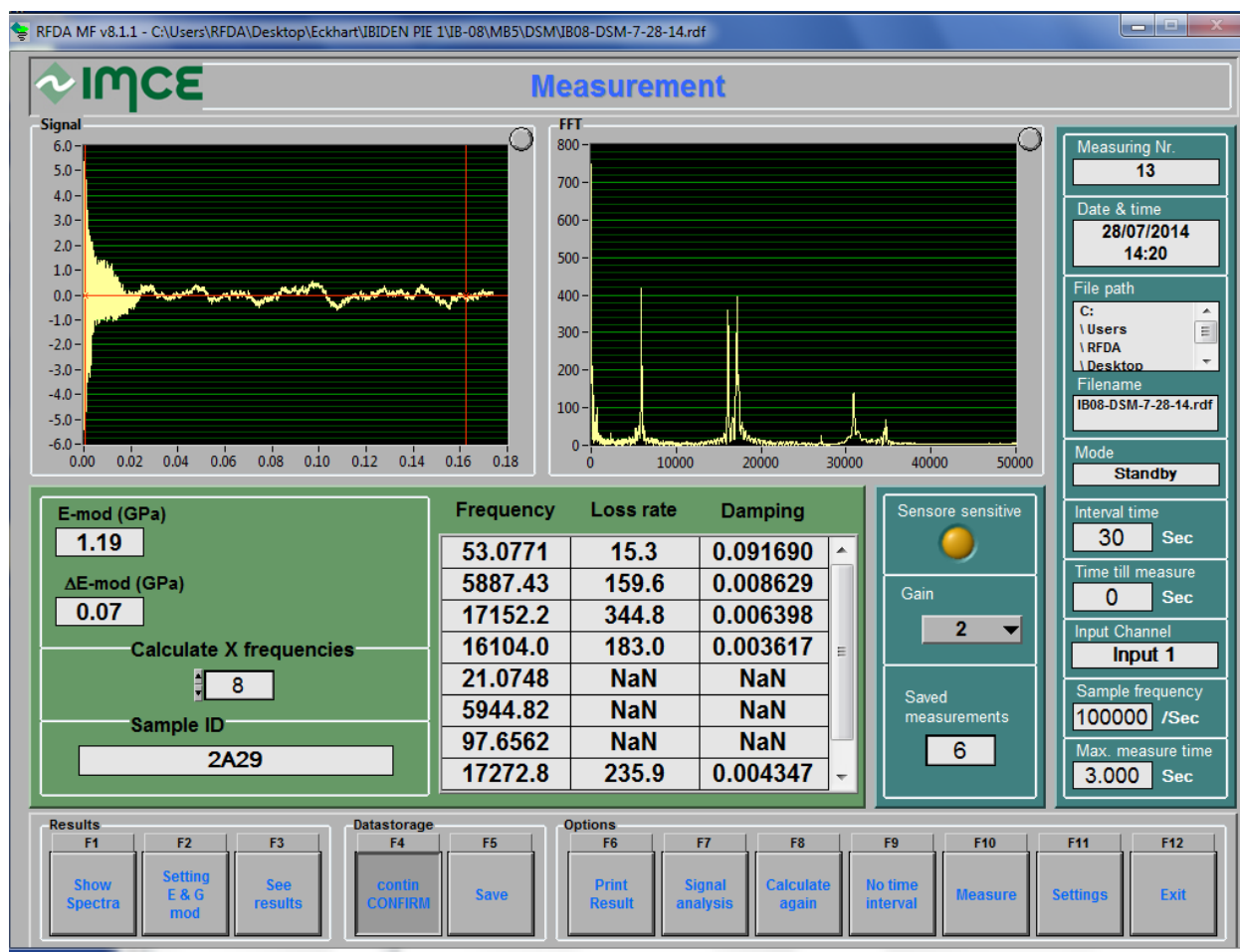


Fig. 5. Screen shot of the IMCE RFDA analysis software GUI. The top-left plot gives the signal recorded by the microphone, while the top-right plot shows the results of the Fourier analysis of the signal. The list of analyzed peaks and the calculated modulus are given in the boxes below the plots.

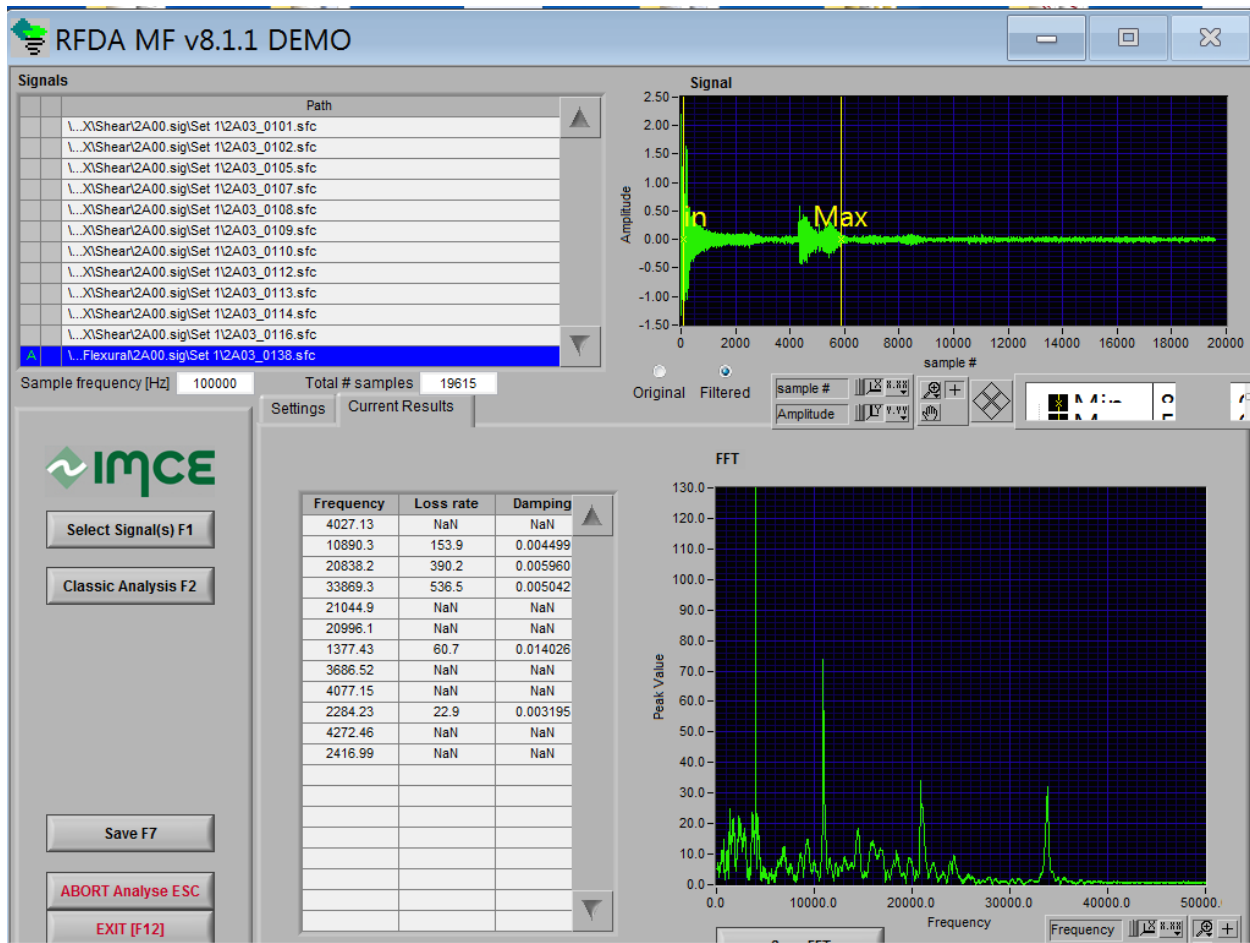


Fig. 6. Screen shot of the RFDA post-processing analysis GUI, showing the results of a flexural measurement. The top plot is the recorded signal, the lower plot is the Fourier analysis, and the peak report is given in the center. Unfortunately for the post-process analysis the software does not calculate the modulus so the values must be calculated *ex-situ*.

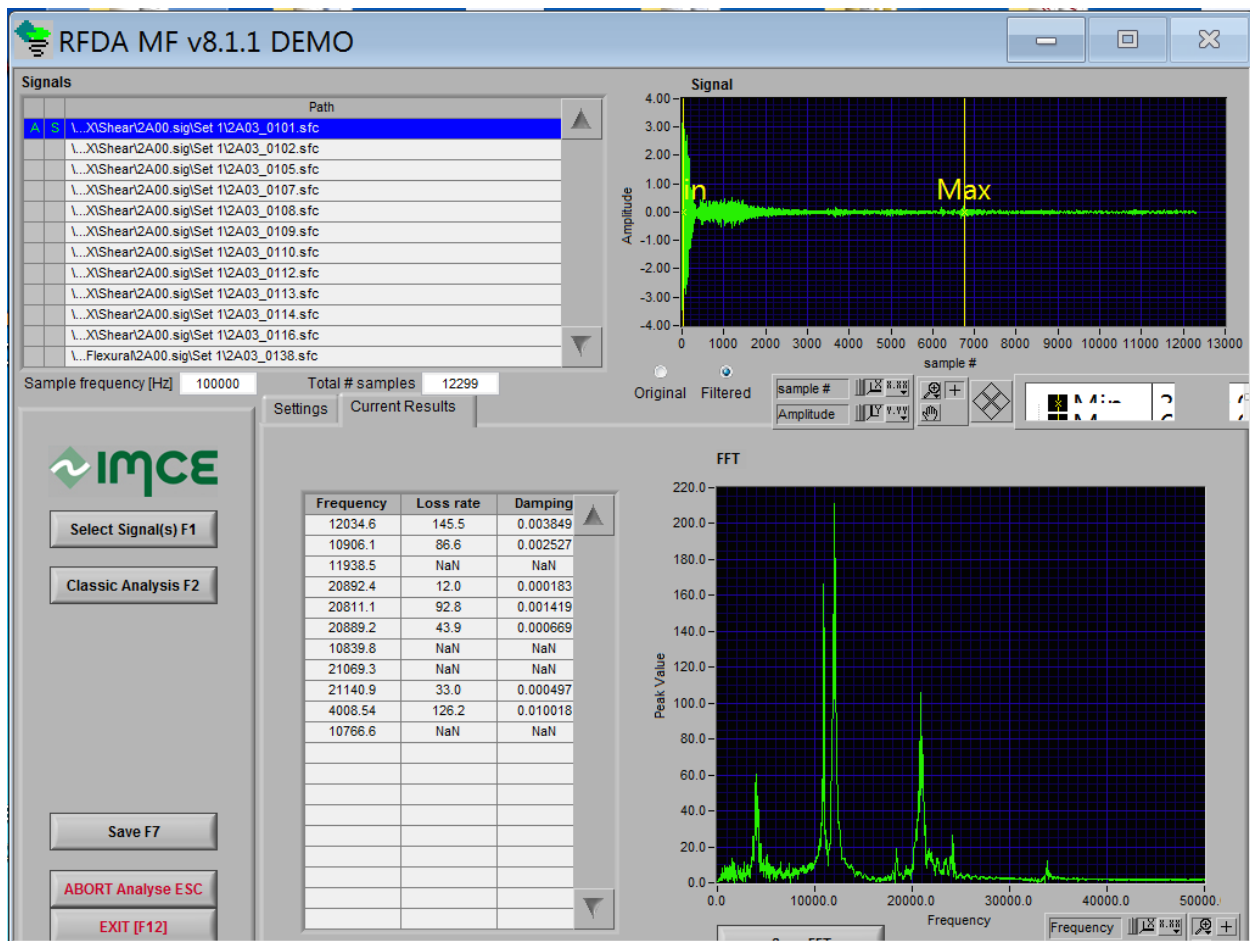


Fig. 7. Screen shot of the RFDA post-processing analysis GUI, showing results of a dynamic shear modulus measurement. This measurement was for the torsional configuration of the same specimen used for the Young's modulus spectrum showed in Fig. 6.

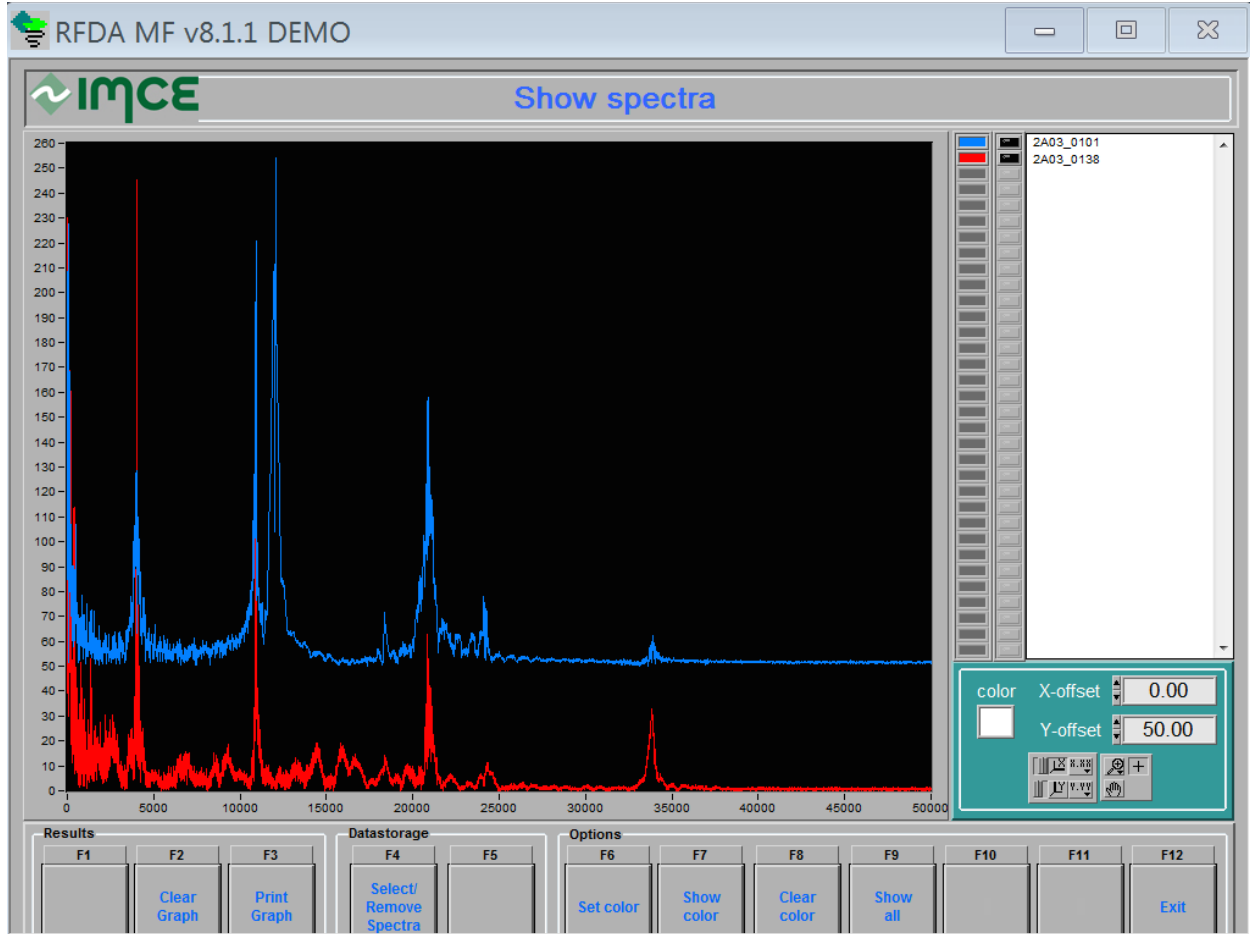


Fig. 8. Screen shot of the RFDA spectrum plotting GUI, showing a dynamic Young's modulus (red spectrum, same spectrum as shown in Fig. 6) and dynamic shear modulus (blue spectrum, same spectrum as shown in Fig. 7) measurements from the same specimen. The activation of the flexural vibration during the torsional setup is the cause of flexural peaks also occurring in the torsional configuration.

6. POTENTIAL ERRORS IN MEASURING SHEAR MODULUS

Certain specimen dimensions can cause a problem to arise in the measurement of the shear modulus, where the fundamental torsional peak and a resonance of the flexural peak to occur at similar frequencies. The Elastic Modulus Good Practice Guide from the National Physical Laboratory in the United Kingdom [4], provides a comprehensive discussion regarding the measurement of the elastic modulus values, including both traditional elastic constant methods and dynamic methods. The section regarding the dynamic methods is of importance for this work, and will be summarized here regarding how it applies to the measurements being performed at ORNL.

Along with altering the harmonic ratios, the thickness to length ratio also changes the ratio of the torsional fundamental frequency and the flexural fundamental frequency. As the thickness to length ratio increases, the ratio of the torsional and flexural frequencies decreases. Additionally, if the thickness to width ratio increases, while keeping the thickness to length ratio constant, the torsional to flexural ratio will increase. An example of this is shown in the plot, reproduced from [4], in Fig. 9. The curves with exponential decay-type behavior show the effect of increasing thickness/length on the torsional/flexural ratio, with the different curves being different thickness/width ratios, and the near-horizontal lines show

the ratios of the harmonics and fundamental flexural frequency. The reason these two behaviors are important is that when the harmonic ratio and torsional/flexural ratios are similar there can be overlap of the peaks during the torsional measurements, which could result in incorrectly measuring the flexural harmonic instead of the torsional peak. Summaries of the thickness/width and thickness/length ratios that cause overlap are listed in Table 4. For example, a specimen with a thickness of 0.5 mm, width of 5 mm, and length of 25 mm ($t/w=0.1$, $t/l=0.02$) would have an overlap between the torsional peak and the 2nd harmonic of the fundamental flexural peak.

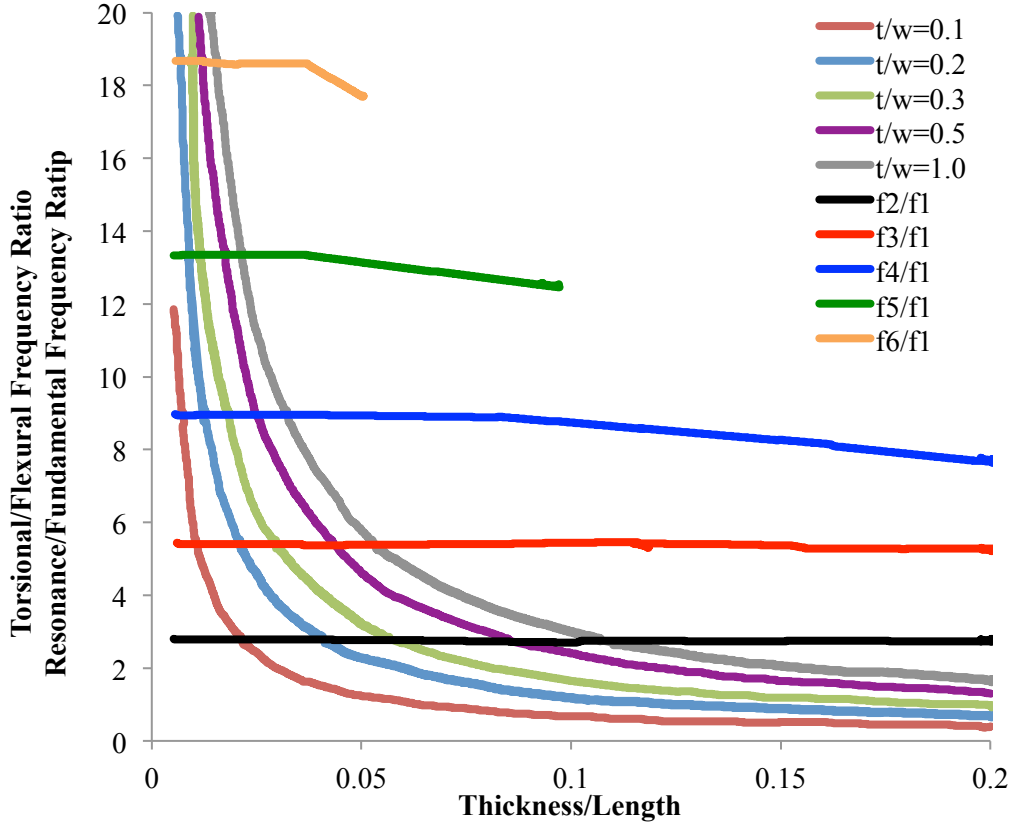


Fig. 9. Plot of the effects of thickness/length, and thickness/width effects on the torsional/flexural and flexural harmonic ratios, from [4]. The lines with exponential-decay shapes are the ratio of torsion/flexural frequencies, with varying thickness/width ratios. The other lines are the ratios of the various flexural harmonics to the fundamental flexural frequency.

Table 4. Summary of thickness/width and thickness/length ratios that cause overlap of the torsional and fundamental harmonic peaks, from [4].

Thickness/Width	Thickness/Length
0.1	0.007, 0.010, 0.020
0.2	0.012, 0.020, 0.043
0.3	0.017, 0.027, 0.055
0.5	0.024, 0.042, 0.084
1	0.031, 0.055, 0.105

7. COMPARISON OF MEASUREMENT RESULTS

Currently, a combination of nine different specimen shapes and five graphite grades, have been measured with both the *GrindoSonic* and the IMCE RFDA systems. Each specimen was measured with a minimum of ten strikes. A summary of the results of these measurements is listed in Table 5, where the listed values are the average and one standard deviation. Overall these two systems showed an average agreement within 1-2%, except for the Ibiden 24 mm x 5 mm x 1 mm and the NTC 48mm x 6mm x 1mm shear modulus specimens. Both of these significantly large disagreements are results of the *GrindoSonic* analyzing the second harmonic of the flexural resonance peak, rather than the torsion peak. This was double checked by calculating the shear modulus using the frequency of the second harmonic, and the resulting shear moduli had agreement with the *GrindoSonic* that again fell within the 1-2% range. An example of the flexural (red) and torsional (blue) spectra, for an Ibiden MB5 specimen, is shown in Fig. 10.

Table 5. Comparison of the Young's and shear modulus values measured by the *GrindoSonic* (E_{GS} and G_{GS}) and the IMCE RFDA (E_{RFD} and G_{RFDA}), for the nine specimen shapes and four graphite grades.

Specimen dimensions (mm)	Specimen shape ID	Manufacture	Graphite grade	Number of specimens	$(E_{GS}-E_{RFD})/E_{GS}$ (%)	$(G_{GS}-G_{RFDA})/G_{GS}$ (%)
30 x 3 x 2.6	SB3	Ibiden	ETU-10	1	0.12	-
24 x 5 x 1	MB5	Ibiden	ETU-10	42	-0.88 ± 1.53	-21.61 ± 4.39
50 x 4 x 2	YMFS	Ibiden	ETU-10	1	-0.31	-
75 x 15 x 3 ^a	SMFS	Ibiden	ETU-10	20	0.14 ± 0.53	0.07 ± 0.46
30 x 3.8 x 2.9	SB-W4	Tokai	G347A	19	3.03 ± 1.14	-
48 x 6 x 1	MB-W6	Tokai	G347A	20	0.69 ± 0.523	-0.39 ± 0.41
50 x 4 x 2	E-FS	Tokai	G347A	1	-1.75	-
70 x 15 x 3	MB-FS	Tokai	G347A	1	-0.07	-0.17
25 x 3 x 2.9	MB	Toyo Tanso	IG-110	55	0.16 ± 0.94	-
25 x 3 x 2.9	MB	Toyo Tanso	IG-430	30	0.01 ± 0.16	-
24 x 3.5 x 2.25	CB	NTC	IGS-743H	20	-0.27 ± 0.33	-
48 x 6 x 1	MB	NTC	IGS-743H	16	1.16 ± 0.46	5.77 ± 5.95

^a Dimensions recommended in ASTM C1198 [2]

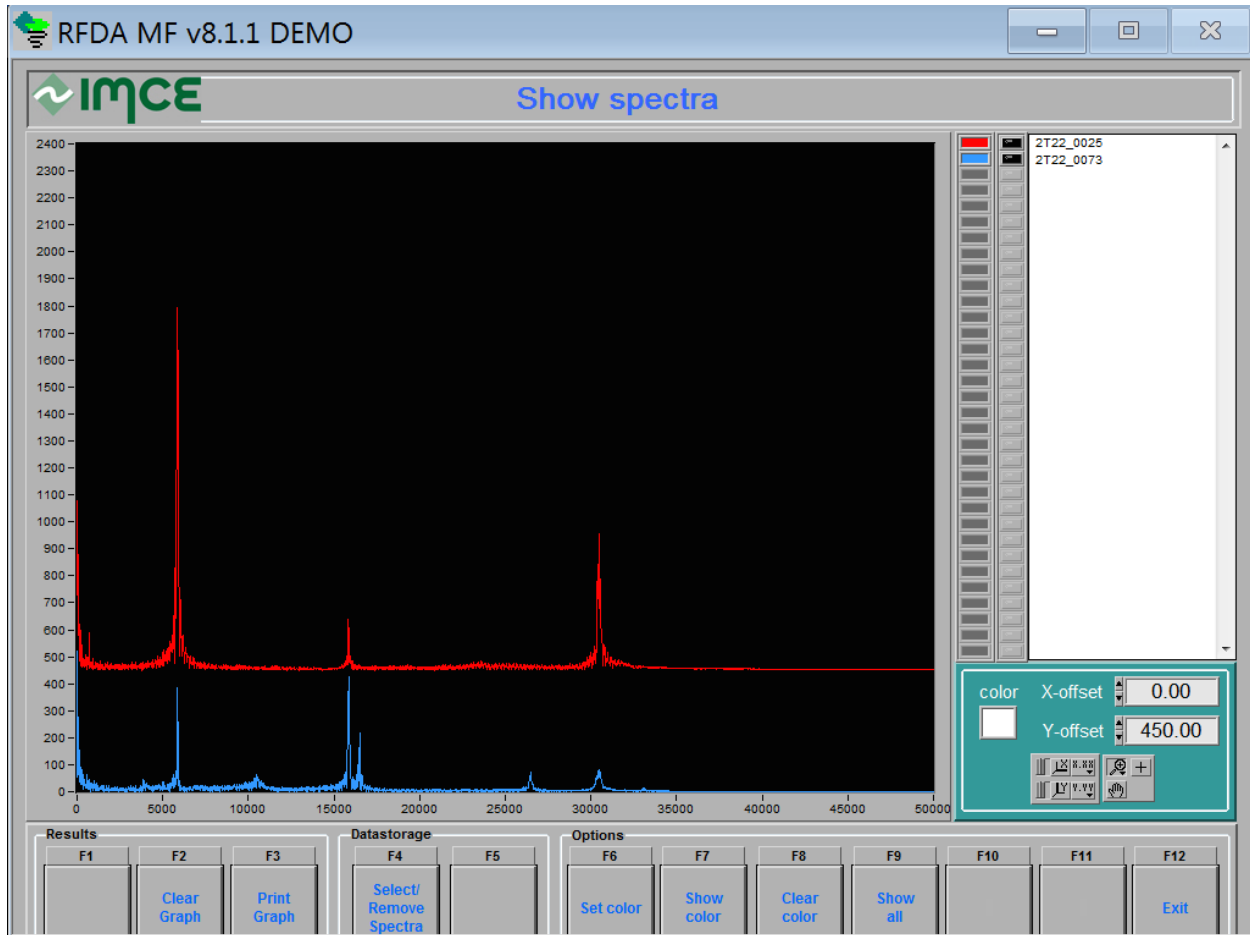


Fig. 10. Dynamic Young's modulus (red spectrum) and dynamic shear modulus (blue spectrum) measurements from the same specimen.

The most-probable cause of the difference in the shear modulus measurements is due to the limited information from the GrindoSonic system, which only outputs the “purest waveform” as the result. If the “purest waveform” does refer to the peak with the highest intensity (within a small frequency range) then it is possible that the values from the GrindoSonic were erroneous for this reason. For the Ibiden specimen, the results from the IMCE RFDA were more vigorously analyzed to determine if this may have been the case with the GrindoSonic. For unadulterated specimens, the torsion peak had larger amplitude than the resonance peak over 90% of the time, 400 times out of 440 strikes on 42 specimens (specimens with average listed in Table 5), but for specimens that have been exposed to neutron irradiation, the torsional peak only had a larger amplitude less than 60% of the time (119 times out of 201 strikes on 20 specimens). An example of this inconsistency of the torsional peak having higher amplitude is shown in Fig. 11, for 11 different strikes for the same specimen, where the red, blue, green, grey, pink, and white spectra show the flexural harmonic peak (around 15500Hz) amplitude is higher than the torsional peak (around 16000Hz). Unfortunately this still doesn't provide an explanation of why the GrindoSonic was outputting the resonance peak frequency rather than the torsional peak frequency.

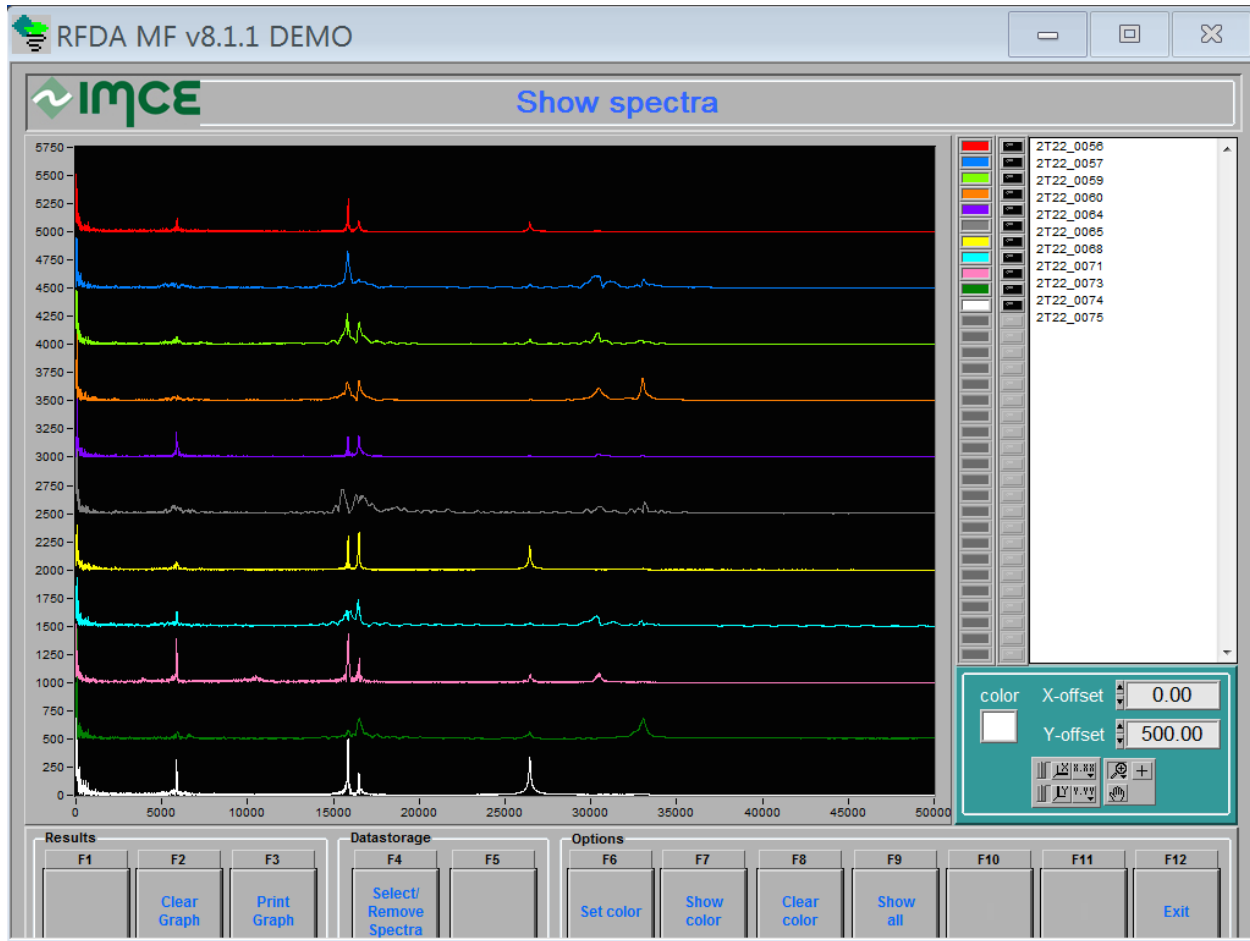


Fig. 11. Eleven spectra collected from multiple torsional measurements on the same specimen. The two peaks between 15000 Hz and 17000 Hz are the flexural resonance (~15800 Hz) and the torsional fundamental frequency (~16500 Hz).

8. SUMMARY

The IMCE RFDA system has recently been purchased to replace the GrindoSonic system, for measuring the dynamic Young's and shear moduli. This system is a preferable method for measuring these properties, primarily for the added control the user has for determining which peaks correspond to the correct peaks for the specific modulus, but also because the user can revisit the measured data when questions arise after the measurements are complete. This is especially crucial for the measurements of shear modulus, where the fundamental shear peak is sometimes near one of the flexural resonance peaks, or when one of the flexural resonance peaks is higher amplitude than the fundamental torsional peak. An example of this was already shown for 24 mm x 5 mm x 1 mm Ibiden specimens, where the GrindoSonic results were from measuring the resonance peak, thereby resulting in a shear modulus that was 20% smaller than the correct value. This new system requires some initial training on setting up the different measurements and using the software, but after training and implementation of a standard operating procedure, the measurement and data analysis process has been greatly improved.

9. REFERENCES

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