



IETeasy: An open source and low-cost instrument for impulse excitation technique, applied to materials classification by acoustical and mechanical properties assessment.



Nazareno Massara^a, Enrico Boccaleri^a, Marco Milanese^a, Mattia Lopresti^{a,*}

^a *Università del Piemonte Orientale, Dipartimento di Scienze e Innovazione Tecnologica, Viale T. Michel 11, 15121 Alessandria, Italy*

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ABSTRACT

In the past twenty years, impulse excitation technique (IET) has become a widely diffused non-destructive technique in metal industry field. This success resides in its capability to determine with high precision and accuracy some elastic properties of materials, such as Young's modulus, shear modulus and Poisson's ratio. The technique, which is very fast and non-destructive, consists in exciting a sample by a mechanical input and registering the acoustic output that, once analyzed by Fast Fourier-Transformation (FFT), provides the resonant frequencies of the sample, with a fast data analysis procedure. The approach is thus very easy to be applied to most materials and cost and time effective. Despite these many advantages, IET is still an under exploited technique in academic research centres, that mainly rely on traditional destructive methods for the evaluation of such properties, for instance by the measurement of strain-stress curves. Commercial IET instruments, similarly to traditional ones, have costs spanning from many hundreds to thousands of dollars, limiting their diffusion in academic world but also in small companies with limited R&D or quality control expenses. Non-professional instruments can also give very precise results and can be successfully used in basic research and in quality control even if not certified as commercial ones. Moreover they can be easily customized according to specific user needs and sample features. Since no examples of low cost IET designs can still be found in the scientific literature, we fill the gap in this paper, giving instructions for a self-assembled instrument for IET analysis, with a cost in the range of 70–85 USD. Moreover, the collected calibration data are analyzed to prove that the instrument can be used for other purposes than the common elastic properties determination, but also for a fast and cheap material characterization exploiting a multivariate analysis approach. Calibration results show that IETeasy can be used in both academic and industrial field for quality control purposes as a low-cost, fast and efficient alternative to tensometers. Principal component analysis, applied in this paper for the first time to IET data analysis, was able to distinguish and classify steel from Al or Cu alloys from polymers, but also different steel grades, demonstrating its potential in massive and eventually automatic IET data analysis. Calculated mechanical properties fitted with good approximation the ranges expected for each sample.

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* Corresponding author.

E-mail address: mattia.lopresti@uniupo.it (M. Lopresti).

Hardware name	IETeasy
Subject area	<ul style="list-style-type: none"> • Engineering and Material Science • Educational Tools and Open Source Alternatives to Existing Infrastructure
Hardware type	<ul style="list-style-type: none"> • Measuring physical properties and in-lab sensors • Mechanical engineering and materials science
Open source license	CC-BY 4.0
Cost of hardware	Approximately 70 USD for the home-made version, approximately 85 USD for the material and additional 50 USD of machine time for the 3D-Printed version.
Source file repository	https://doi.org/10.17632/c8sxdwds5x.1

1. Hardware in context

In the last years of the 1990s, [1,2] Impulse Excitation Technique (IET) was proposed as an easily applicable method for the measurement of resonant frequencies of materials, which can be exploited to calculate elastic properties, such as Young's modulus, shear ratio and Poisson's coefficient. The approach can be thought of as playing a percussion instrument, which consists in generating a mechanical impulse in a sample, using a mallet or a projectile, and collecting the audible output sound-wave. The data collection can be performed by different sensors depending on the availability: microphones, accelerometers, piezoelectric sensors, or laser vibrometers. Then, data are processed usually by FFT, which converts the sound-wave signals in the time domain to spectral data in the frequency domain. In the obtained spectra, natural resonant frequencies of the samples are identified by the peak positions and, by applying Euler-Bernoulli beam theory for Free-Free boundary conditions [3], elastic properties of materials can be determined. Despite being a relatively simple approach, the accuracy of the technique is very high, and the uncertainty on the calculated moduli can be lowered to 0.1% [4]. IET is also very versatile and can be used in a wide range of temperatures (from $-50\text{ }^{\circ}\text{C}$ to $1700\text{ }^{\circ}\text{C}$ [2]) and in presence of different gases or in vacuum, depending on which sensor is equipped on the instrument (laser vibrometers is necessary for vacuum measurements). In the 2000s, the industrial world implemented this approach in the quality control processes, while regulatory bodies [5–7] provided technical standards for IET procedures. Despite its capability to provide precise and accurate results in a very short time, IET initially remained mainly confined in the industrial world and used for quality control purposes. However, even in this field, other expensive complementary techniques had been preferred to IET, such as the ultrasounds analysis, radiography and tomography [8], which are still the most used non-destructive techniques (NDT) for quality control and certification. In the last years, many notable academic research examples began to spread in the fields of the metallurgy [9–11], geology [12] and the science of ceramic materials [13–15]. Even if IET data are rich in information content, the main application of the technique consists in the measurement of elastic properties of materials, such as Young's modulus, shear modulus and Poisson's coefficient [16]. In some research articles, IET is used on samples to detect and study the nature of defects [17,11], stress [18] and voids [19]. The technique can be used, not only for the elastic characteristics of a material and its damping properties, but also for material classification and qualitative analysis of samples. For these purposes, multivariate statistical analysis can cover a key role with many unexplored and promising applications, with the most common pattern recognition and classification methods (Cluster analysis, principal component analysis, linear discriminant analysis) being used for sample identification. In more complex and wider data collections, machine learning can be also used to build an automated recognition system. These applications are still scarce in the scientific literature and this can be ascribed to two concurrent causes. On one hand, many professional instruments are often rather costly, especially the portable ones and do not allow the full analysis of the collected impulses due to factory settings or for lacks of suitable algorithms. In particular, some instruments are designed to give the Young's module of sampled materials as output, which is only one of the responses that can be extracted by a complete analysis of the impulse. Academic research requires a direct control and handling of raw data. On the other hand, despite being known as a technique that does not require a professional equipment, in scientific literature, no examples of low-cost and/or open source instruments for IET can be found. To overcome these obstacles hindering a wider diffusion in academic world, but also in small companies not able to afford a professional instrument, a self-built instrument for IET analysis (IETeasy) is presented in two different versions: one built in spruce wood and another one, identical in shape but in polylactic acid (PLA), made by 3D-Printing. The main difference between the two setups resides in their aesthetic, with the 3D-printed version being more elegant than the spruce wood one. However, the 3D-printed version, can be reproduced more easily in many identical exemplars. Some samples of different materials were then analyzed with the IETeasy and both raw and processed data are provided in an online Mendeley Data repository [20] and described in the respective data article [21]. Descriptive statistics on the collected data and a demonstration of the capability in materials recognition are provided in Section 7. Young's moduli for these materials were calculated and compared to the expected ones reported in literature or in technical data sheets. Finally, the potentialities of principal component analysis (PCA) in fast, efficient and eventually automatic analysis of very large data set by IET spectroscopy is demonstrated, since PCA can be a con-

venient complementary or alternative approach to Fourier transform based methods, as demonstrated in crystallography field by some of us [22].

2. Hardware description

IETeasy is an instrument very simple in its design, composed by two separate parts: a sample-holding frame and a mallet support. The first part is a frame with two taut nylon strings with diameter of 0.5mm. The strings have the task of supporting the sample and, at the same time, implementing Free-Free boundary conditions as well as possible by placing them along the nodal lines of the desired vibration mode. Inside the frame, a passing bar is placed with a centimeter scale, used to measure the distance between the two strings and guarantee that all the data collections are done in the same conditions. The centimeter scale is, not only convenient to guarantee that data are collected in the same conditions, but also to place the strings along the nodal lines. Moreover, the central bar has the task of supporting the strings, in order to avoid distortion effects that can be caused the string vibrations against flat surfaces of the frame. The mallet support consists in a xylophone mallet mounted on a wooden standing support which has the task of preventing differences in the exciting impulse strength. Being only gravity-based, the hitting strength of the mallet is always the same, which guarantees that the measurement as reproducible as possible. A USB condenser microphone connected to a PC is used as acquisition setup. The head of the microphone is positioned above the sample and directed toward its upper side, as displayed in Fig. 1. An average acquisition spans between 8 and 12s, depending on how much damped is the sound produced by the sample.

2.1. Traditional data analysis methods

The collected raw data are in the time domain and they can be used for the calculation of the damping parameter of the investigated samples, which gives an estimation of the material's behavior in response to an induced oscillation. It has to be considered that the calculation of the acoustic insulation can not be accurate with this setup, as the air viscosity it's usually not negligible. Vacuum chamber and a laser vibrometer (or another non-acoustic sensor) are required for a precise measurement. Traditional analysis can be carried out on both time or frequency domain. The calculation can be done by refining the damped sound wave with an exponentially damped sine function in time domain in the form [23]:

$$s_t = Ae^{-kt} \sin(2\pi f_r t + \phi) \quad (1)$$

where:

1. s_t is the collected sound wave in the time domain;
2. A is a scale coefficient that have to be refined;
3. k is the decrease parameter due to the damping;
4. t is the time;
5. f_r is the frequency of the fundamental flexion vibration mode;
6. ϕ is the wave phase.

Often, such refinements are not easy to perform due to the superposition of several modes. The time domain data are then processed by FFT to transform the signal in the frequency domain and extract the resonant frequencies, which can be used for qualitative analysis or to calculate the mechanical properties, or even to determine if a sample has structural defects. For mechanical properties, on rectangular samples (the ones for which this instrument is designed), Young's modulus (E) of rectangular samples can be calculated as [5,7,6]:

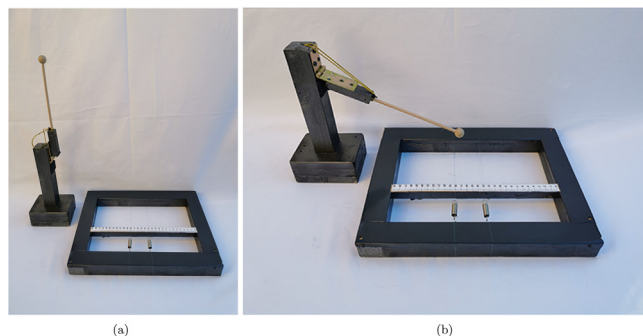


Fig. 1. One of the wooden self-built version of IETeasy instrument.

$$E = 0.9465 \left(\frac{m^2 f_r^2}{w} \right) \left(\frac{l^3}{t^3} \right) C \quad (2)$$

Where:

1. m is the mass in kilograms of the sample;
2. f_r is the frequency of the fundamental flexion vibration mode;
3. w is the width in metres of the sample;
4. l is the length in metres of the sample;
5. t is the thickness in metres of the sample;
6. C is the correction factor defined as:

$$C = 1 + 6.585 \left(\frac{t}{l} \right)^2 \quad (3)$$

The correction factor should be used only when $l/t \geq 20$.

2.2. Multivariate data analysis as a fast and efficient approach to IET data

Multivariate analysis allows a precise qualitative analysis and, differently from traditional data analysis, can be performed for classification purposes on both time domain and FFT-processed frequency data. In other fields, it resulted a very efficient alternative or complementary technique to FFT based method, as demonstrated by some of us in crystallography [22,24]. One of the main advantages of using a multivariate approach consists in the reduction of dimensionality, in which useful information is extracted from a large set of variables and rewritten in a few “easy to interpret” variables. More important is its capability of easily, fastly and very efficiently analysing very large dataset also in an automatic fashion. In this paper we demonstrated PCA huge potentialities in IETeasy data analysis, and in general, on IET data. Consequently, a fast data analysis coupled to a tool that can produce a huge amount of data in a short time is suitable for on-line analysis (i.e. waste disposal centres). Of course no direct advantage is evident in the small training set used in this paper, but PCA can be easily applied to thousands of data set in a few time and also in an automatic fashion, differently from traditional analysis methods, using mostly (or only) the fundamental frequency. An example of this approach is given in Section 7.3 with the principal component analysis applied to the validation of the data. Also, by measuring series of samples with the same size and shape, it is possible to determine whether defectivity or deviation from conformity is present in a sample or not, making an automatic version of this instrument a good candidate for an on-line quality control.

- Mechanical properties can be determined with high precision and accuracy on materials of different natures;
- Classification analysis can be performed by using both time domain and frequency domain data. Samples can be very well distinguished in the corresponding classes with a highly precise approach;
- By calculating the damping coefficient, a rough estimation of the acoustic insulation of the material can be performed. The use of a vacuum chamber and a laser vibrometer might improve the precision of the calculation, but such approach is beyond the scope of the present contribution.

3. Design files

For the wood version of the IETeasy, detailed building instructions and illustrations are reported in Section 5, therefore no design files were produced. For the 3D-Printed version of the instrument, the required `.stl` files can be found as [Supplementary material](#) of the present article. All files are detailed in the following table.

3.1. Design files summary

Design filename	File type	Open source license	Location of the file
angle a	stl 3D file	CC BY-NC-ND 4.0	Available within the repository
angle b	stl 3D file	CC BY-NC-ND 4.0	Available within the repository
base a	stl 3D file	CC BY-NC-ND 4.0	Available within the repository
base b	stl 3D file	CC BY-NC-ND 4.0	Available within the repository
central bar	stl 3D file	CC BY-NC-ND 4.0	Available within the repository
canti	stl 3D file	CC BY-NC-ND 4.0	Available within the repository
pin a	stl 3D file	CC BY-NC-ND 4.0	Available within the repository
pin b	stl 3D file	CC BY-NC-ND 4.0	Available within the repository

4. Bill of materials

4.1. Bill of the wood structure

Designator	Number	Cost per unit currency	Total cost	Source of materials	Material type
Wood strip 40 mm × 40 mm × 1 m	3	4.04 USD per piece	12.12 USD	Leroy Merlin	Spruce wood
Wood strip 20 mm × 20 mm × 1 m	1	2.49 USD per piece	2.49 USD	Leroy Merlin	Spruce wood
Vinyl glue	1	2.40 USD per 225 g	2.40 USD	Leroy Merlin	Vinyl glue
Wood screw 6 mm × 4 mm	7	14.70 USD per kg	0.25 USD	Leroy Merlin	Bronzed steel

4.2. Bill of the 3D-Printed structure

Designator	Number	Cost per unit currency	Total cost	Source of materials	Material type
angle a	2	35.69 USD per kg	11.64 USD	Aliexpress	PLA filament, $\phi = 1.75$ mm
angle b	2	35.69 USD per kg	11.64 USD	Aliexpress	PLA filament, $\phi = 1.75$ mm
base a	1	35.69 USD per kg	4.39 USD	Aliexpress	PLA filament, $\phi = 1.75$ mm
base b	1	35.69 USD per kg	2.74 USD	Aliexpress	PLA filament, $\phi = 1.75$ mm
central bar	1	35.69 USD per kg	1.64 USD	Aliexpress	PLA filament, $\phi = 1.75$ mm
canti	1	35.69 USD per kg	0.36 USD	Aliexpress	PLA filament, $\phi = 1.75$ mm
pin a	3	35.69 USD per kg	0.86 USD	Aliexpress	PLA filament, $\phi = 1.75$ mm
pin a	4	35.69 USD per kg	0.22 USD	Aliexpress	PLA filament, $\phi = 1.75$ mm

For the 3D-printed version of the frame and the mallet support, the required total estimated time is of ≈ 100 h of machine time. For an average 3D-printer, the electric consumption is about 70W for an hour, using a heated bed temperature of 60°C and an hotend temperature of 205°C. Therefore, the costs of the 3D-printing process can not be ignored and are estimated as 50 USD, depending on the cost of 1 kWh and common maintenance costs.

4.3. Bill of the essentials

Designator	Number	Cost per unit currency	Total cost	Source of materials	Material type
Nylon wire 0.5 mm × 100 m	1	5.91 USD per 100 m	5.91 USD	Leroy Merlin	Nylon
Electrician clamp	2	2.77 USD 12 pieces	0.46 USD	Amazon	Plastic and brass clamps
Xylophone mallet	1	9.15 USD 4 pieces	2.29 USD	Amazon	Wood mallet
USB condenser microphone	1	40.23 USD per piece	40.23 USD	Amazon	Composite
Flat hinge 25 mm	1	2.59 USD per piece	2.59 USD	Amazon	Steel

5. Build instructions

The dimensions and materials of the instrument have been arbitrarily chosen by the authors, based on the size of the samples that are usually analyzed in the laboratory work scale. Other materials and sizes can be chosen as needed. In particular, the mallet support is an optional component of the instrument as it was observed in the testing phase that even free-hand data impulses have excellent reproducibility on the position of the resonant frequencies. Mallet support has been incorporated into the basic design of the instrument as it helps standardize the intensities of spectral signals, which would otherwise be subject to high variability.

5.1. Frame and sample-holding strings

In this section, the “made in wood” IETeas building instructions are reported. Concerning the corresponding PLA 3D-Printed version, the single pieces have unequivocal interlocks, thus no specific instructions are needed for assembling the PLA frame and the mallet support. Once the PLA frame is built, the operations for mounting the wires are the same of the wood version, therefore only instructions from point 4 onward are required for the 3D-Printed IETeas version.

1. Two square stripes of spruce wood, one larger with section size of 4 cm, and one smaller with section size of 2 cm were cut to obtain the four frame pieces and the passing central bar that will hold the wires in tension as showed in Fig. 2(a). The larger strips are cut 40 cm in length, while the smaller strip is cut 32 cm long.
2. To avoid wood fissuring along its natural venatures, pre-holes for wood screws were made on the four corners before screwing together the four strips of the frame as shown in Fig. 2(b). The pre-holes were made using a battery drill with a 3.5 mm in diameter drill bit. The central bar was fixed with wood screws inserted in pre-holes as shown in Fig. 2(c). Every surface irregularity can be removed by using sandpaper for a better aesthetic result.
3. On the central bar a centimetre scale was glued (Fig. 2(d)) in order to have a reference of the distance between the sample-holding wires. It is not important that the centimetre scale is positioned at the beginning of the central bar, because it's unlikely that wires will be separated more than 20 centimetres due to possible collisions between the sample and the frame. If wanted, rubber feet can be attached to the lower part of the frame to keep all the instrument suspended from the underneath table for a better aesthetic result. As spruce wood is soft, the wire can indent the surface, metal plaque can be added to preserve the structure as displayed in Fig. 1.
4. Two pieces of 1 m each of nylon wire with diameter 0.5 mm was cut to make the sample holder. The wires are passed around both the central bar and the opposite side of the frame, as shown in Fig. 2(f). In the version showed in Fig. 2, wires are passed through two holes at the desired measurement distance. Optionally, the wires can be kept in tension using two steel springs, as shown in Fig. 1, which let wires to be custom-positioned at each measurement.
5. The two wires are then taut and kept in position by using electrician clamps. The wire tension can be checked by using a guitar tuner. In this case, an A₄ note was used to accord the wires, but this specific note is not necessary to obtain reproducible measurement: the important check is conversely having both wires equally taut and giving the same note, in order to avoid undesired damping effects on the sample.

5.2. Mallet support

Similarly to what stated for the frame, the 3D-Printed version of the mallet support does not require instructions as interlocks are unequivocal. Only flat hinge and mallet instructions have to be read, from the following point 3 onward.

1. Three pieces 12 cm long and 4 cm wide are glued together using vinyl glue in order to obtain a squared piece that will act as the base of the mallet support. Once the glue is set, surface irregularities can be removed by using sandpaper (Fig. 3(a)).
2. A strip 25 cm long and 4 cm wide and a strip 7 cm long and 2 cm wide are cut and will be used as stand part and mallet support respectively.
3. A pre-hole is made at the center of the base and a strip in order to fix by using a screw the 25 cm long piece as shown in Fig. 3(b). To avoid rotations of the vertical part, applying some glue is suggested.
4. A hole 1 cm deep, with a diameter slightly smaller than the one of the mallet strip is made on the flat part of the support as displayed in Fig. 3(c). The piece is fixed to the vertical mallet support with the hinge in order to have a semicircular movement toward the bottom. The mallet is glued and fixed in the proper hole as shown in Fig. 3(d).
5. A rubber band is used to keep the mallet suspended after the impulse is given. There is no particular indication about the rubber band, because it has to be evaluated by the user depending on the setup positioning. The authors used a standard commercial rubber band with a section of 1 mm × 5 mm and a diameter of 42 mm.

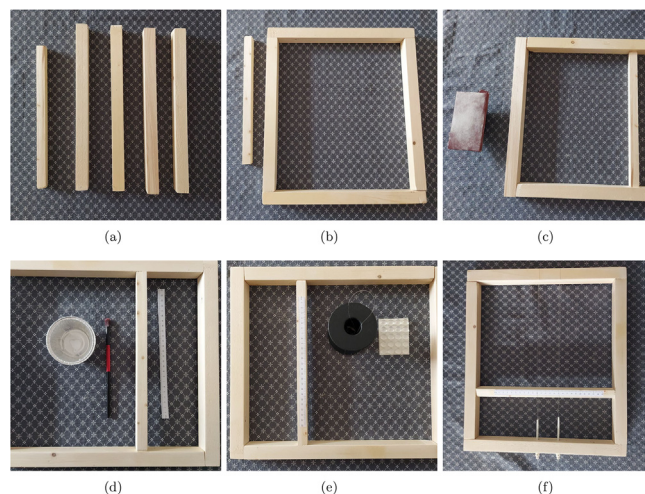


Fig. 2. Visual building instructions of the sample-holding frame.

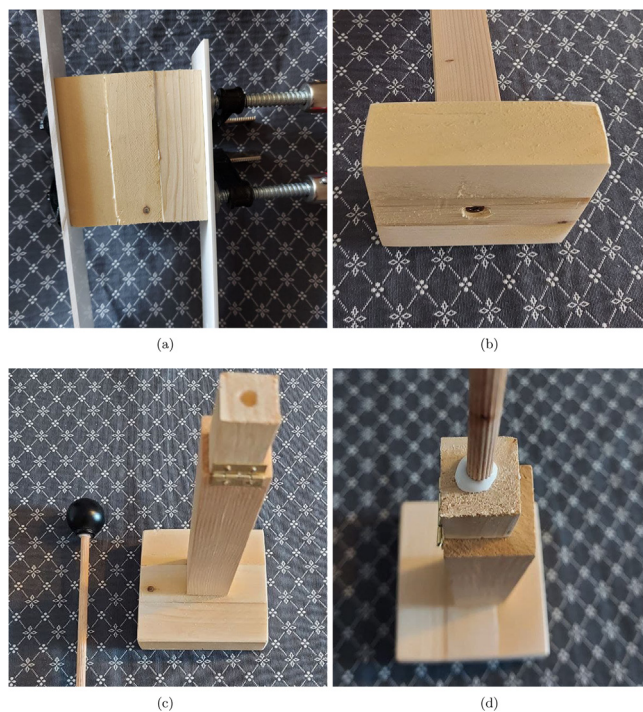


Fig. 3. Visual building instructions of the mallet support.

6. Operation instructions

- Place the instrument on a flat stable surface, with the microphone positioned as shown in Fig. 4. Check the distances between the frame and the mallet support and be sure that the mallet can hit the sample correctly once positioned.
- Connect the USB microphone to the PC used to record the impulse. Open the recording software and check that the USB microphone is default input device and that the audio signal is correctly read by the PC. In this paper, Audacity 2.4.2 [25] was used to manage sound sampling.

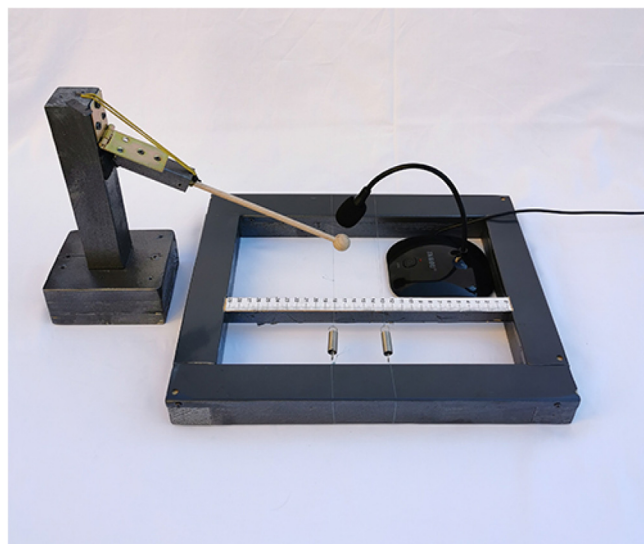


Fig. 4. Acquisition setup. The sample is positioned on the sample-holding strings and the mallet is released, giving the mechanical excitation to the material. The produced sound wave is collected by the microphone positioned over the sample and then is processed for further analyses.

- Check the correct distancing between the sample-holding strings. In this paper, standard distance is 5 cm as samples are about 10 cm long.
- Place the sample on the supporting strings and check that the microphone is correctly positioned over it as shown in Fig. 4.
- Start recording and, after few seconds, release the mallet.
- Stop recording after 5–6 s after the impulse production, when the damping is finished.

7. Validation and characterization

The IETeasy hardware was tested initially in its different components and with different purposes: at first, the uncertainty due to the microphone was tested (Subsection 7.1) by collecting notes generated at given frequencies. Then, real-world samples of fifteen different materials were measured by using IETeasy ten times each, in order to obtain descriptive statistics on the natural resonant frequencies of the materials, using an traditional univariate approach (Subsection 7.2). In this section, Young's moduli had been calculated and compared to the tabulated values on the corresponding technical data sheets for each material. In subSection 7.3, the whole frequency spectra were analyzed with a multivariate approach by using Principal Component Analysis (PCA) to demonstrate its capability in their classification and the potentialities of such an approach when dealing with thousand data files. For the validation of the hardware construction procedure, two different IETeasy instruments were built by two different users (NM and ML) and then massively used, thus suggesting that multiple IETeasy can easily be assembled and used for materials analysis and classification with good reproducibility.

7.1. Microphone distortion

The accuracy of the microphone was tested by putting it in front of a speaker and generating sound waves corresponding to the seven notes on nine octaves, from A_0 to B_8 . Then, those sounds were recorded and processed with FFT to transform the time-domain signals in frequency-domain signals. In Table 1, the generated frequencies and the acquired ones are reported.

As can be seen in Table 1, the errors relative to the acquisition setup are very small and affecting the last one or two significant figures, therefore it can be assumed that the intrinsic overall error is less than the 0.01%, with respect to the measurement itself.

7.2. Characteristic resonant frequencies

Samples of different materials (metals and polymers), were then measured with ten repetitions each, and the collected data were processed by FFT to further analyses reported in Tables 2 and 3. Details on both the samples and the data collection and FFT algorithm are given in the related data article [21]. Obtained spectra were analyzed with the `pick.peaks` function of the `ChemometricsWithR` package in R, in order to extract the principal resonant frequencies. The results are very precise for alloys but then become more noisy for polymers, as their internal structure is very different, not only due to the long polymeric chains that have more degree of freedom, and a consequent less efficient phononic propagation, but also because of their amorphous structure, which causes an internal damping effect. When plotting the original sound-waves, it can be noticed that the acquisition time of polymers is far shorter than those from the alloys, simply because of the different damping of the materials.

By looking at the descriptive statistics in Table 4, calculated on the resonant frequencies of Tables 2 and 3, it can be observed that alloys samples best perform as reproducibility of the method, with a coefficient of variation of $10^{-3} - 10^{-4}$ percent. Polymers, due to their molecular structures, characterized by long interconnected chains and amorphous (or less crystalline) phases, have a greater variation coefficient, about 100 times greater than the ones obtained for metals. Despite this, the coefficient of variation of this material is still very low, being only 0.56%.

Table 1

Musical notes from A_0 to B_8 . For each note the $_{read}$ value is the value obtained by processing by FFT the collected data, while the $_{gen}$ is the note produced by a frequency generator. All data are in Hz.

Octave	C_{read}	C_{gen}	D_{read}	D_{gen}	E_{read}	E_{gen}	F_{read}	F_{gen}	G_{read}	G_{gen}	A_{read}	A_{gen}	B_{read}	B_{gen}
0	–	–	–	–	–	–	–	–	–	–	27.480	27.500	30.879	30.868
1	32.705	32.703	36.722	36.708	41.209	41.203	43.639	43.654	49.014	48.999	55.006	55.000	61.727	61.735
2	65.405	65.406	73.416	73.416	82.435	82.407	87.336	87.307	97.999	97.999	110.02	110.00	123.47	123.47
3	130.86	130.81	146.84	146.83	164.79	164.81	174.60	174.61	196.02	196.00	220.00	220.00	246.95	246.94
4	261.70	261.63	293.75	293.67	329.65	329.63	349.32	349.23	392.03	392.00	440.05	440.00	493.88	493.88
5	523.28	523.25	587.41	587.33	659.30	659.23	698.49	698.46	784.04	783.99	880.14	880.000	987.92	987.77
6	1046.6	1046.5	1174.8	1174.7	1318.6	1318.5	1397.0	1396.9	1568.1	1568.0	1760.3	1760.0	1975.7	1975.5
7	2093.2	2093.0	2349.6	2349.3	2637.4	2637.0	2794.2	2793.8	3136.4	3136.0	3520.4	3520.0	3951.6	3951.0
8	4186.5	4186.0	4699.2	4698.6	5274.7	5274.0	5588.3	5587.7	6272.6	6271.9	7040.8	7240.0	7903.1	7902.1

Table 2

Frequencies of the fundamental flexion vibration mode in Hz for each sampled material. For each sample, 10 impulses were collected.

Sample #	AISI 304 steel	AISI 316 steel	Aluminum 6082	B10 bronze	B12 bronze	BrAl alloy	C45E steel
1	3845.167	4953.186	5171.082	4170.651	4487.253	2892.7	5045.326
2	3845.242	4953.369	5171.082	4170.583	4487.288	2892.654	5045.331
3	3845.200	4953.323	5171.082	4170.607	4487.262	2892.654	5045.390
4	3845.158	4953.278	5171.082	4170.714	4487.337	2892.609	5045.420
5	3845.233	4953.278	5171.082	4170.680	4487.334	2892.609	5045.444
6	3845.104	4953.186	5171.082	4170.622	4487.334	2892.609	5045.374
7	3845.164	4953.278	5171.173	4170.559	4487.361	2892.609	5045.371
8	3845.237	4953.278	5171.127	4170.637	4487.300	2892.609	5045.336
9	3845.153	4953.278	5171.036	4170.652	4487.277	2892.609	5045.406
10	3845.174	4953.278	5171.082	4170.697	4487.312	2892.609	5045.429

Table 3

Frequencies of the fundamental flexion vibration mode in Hz for each sampled material. For each sample, 10 impulses were collected.

Sample #	Copper	CW614 brass	Fe37 drawn	HDPE	Nylon 6	Pom-C	Teflon	X150 steel
1	3635.632	3072.876	4236.647	1520.05	1893.813	1814.667	840.150	5100.128
2	3635.591	3072.922	4236.590	1520.035	1921.683	1812.744	840.857	5100.128
3	3635.602	3072.922	4236.632	1519.958	1904.682	1815.308	839.649	5100.128
4	3635.535	3072.876	4236.697	1520.05	1917.503	1811.92	839.877	5100.128
5	3635.514	3072.876	4236.680	1519.684	1927.536	1811.462	839.375	5100.037
6	3635.520	3072.876	4236.646	1524.628	1907.469	1812.012	841.883	5100.128
7	3635.597	3072.876	4236.635	1522.293	1921.683	1810.822	839.945	5100.128
8	3635.492	3072.876	4236.641	1523.163	1917.503	1811.188	840.629	5100.128
9	3635.514	3072.876	4236.642	1523.071	1928.372	1811.646	841.884	5100.128
10	3635.512	3072.876	4236.679	1519.409	1917.503	1812.469	841.883	5100.137

Table 4

Descriptive statistics of the collected resonant frequencies.

Sample	Mean	Median	Range	Std.Dev.	Coef.Var.
AISI 304 steel	3845.183	3845.171	0.138	0.044284	0.001152
AISI 316 steel	4953.273	4953.278	0.183	0.054776	0.001106
Aluminum 6082	5171.091	5171.082	0.137	0.035920	0.000695
B10 bronze	4170.640	4170.644	0.155	0.049241	0.001181
B12 bronze	4487.306	4487.306	0.108	0.035801	0.000798
BrAl alloy	2892.627	2892.609	0.091	0.031719	0.001097
C45E steel	5045.383	5045.382	0.118	0.042387	0.000840
Copper	3635.551	3635.528	0.14	0.049256	0.00136
CW614 brass	3072.885	3072.876	0.046	0.019395	0.000631
Fe37 drawn	4236.649	4236.644	0.107	0.030289	0.000715
Nylon 6	1915.775	1917.503	34.559	10.801986	0.563844
HDPE	1521.234	1520.05	5.219	1.866040	0.122666
Pom-C	1812.424	1811.966	4.486	1.472017	0.081218
Teflon	840.6132	840.3895	2.509	0.975587	0.116057
X150 steel	5100.120	5100.128	0.1	0.029230	0.000573

Young's moduli of the analyzed materials were determined using Eq. 2. All calculated values are compared to the corresponding average values in Table 5. The reported tabulated values are taken from literature [26] or from specialized web-sites, as no experimental data were reported in technical data sheets given by the supplier. Stainless steel reported values that are about 30 GPa lower than the expected value, while for other materials the difference between the calculated and the expected data is very low.

As a final note on the reproducibility during IETeasy usage and on the stability of the whole hardware and its components, it must be pointed out that the two built instruments were massively used in preliminary analyses and in the campaign to obtain the data described in the Data in Brief article [21]. Hundreds measurements were carried out and resulted reproducible within time, without progressive drifts in the measurements and without changes in the experimental error, also comparing data by the two above-mentioned different instruments.

7.3. Multivariate analysis

PCA is a common method for pattern recognition analysis, which is often used to explore relationships between samples and variables. A detailed description of the method applied to xy data and the interpretation of its results is reported in a

Table 5

Calculated Young's moduli for the analyzed materials. The obtained values are compared to tabulated values from collections of technical data sheets.

Sample	Calculated Young's modulus	Tabulated Young's modulus
AISI 304 steel	166 GPa	193 GPa [26]
AISI 316 steel	170 GPa	193 GPa [26]
Aluminum 6082	69 GPa	67.0 GPa to 70.0 GPa [27,28]
B10 bronze	92.5 GPa	90 GPa to 110 GPa [29]
B12 bronze	93.9 GPa	90 GPa to 110 GPa [30]
BrAl alloy	119 GPa	125 GPa [31]
C45E steel	184 GPa	190 GPa [32]
Copper	132 GPa	118 GPa to 132 GPa [33]
CW614	107 GPa	105 GPa [34]
Fe37 drawn	201 GPa	200 GPa [35]
HDPE	2.16 GPa	0.65 GPa to 4.30 GPa [36]
Nylon 6	4.08 GPa	1.30 GPa to 4.20 GPa [37]
Pom-C	4.29 GPa	0.59 GPa to 11.7 GPa [38]
Teflon	1.19 GPa	0.39 GPa to 2.25 GPa [39]
X150 steel	181 GPa	190 GPa [40]

recent review published by some of us [24], in the field of powder crystallography. The same approach and concepts are here applied to IETeasy data to give an example on how a large collection of samples can be analyzed in a fast and efficient way to obtain a classification. The analysis was performed by using RootProf [41], a software developed at CNR of Bari and based on CERN's ROOT framework.

Collected data were processed by FFT and the resulting sound spectra were sub-sampled and analyzed by RootProf to test the capability in sample recognition for qualitative analysis purposes. Samples and data pre-processing are described in the corresponding article [21]. The sub-sampling consisted in arbitrarily dividing the whole data set in three different batches with different homogeneity concerning sample features. The subset are described as follows in order to submit to the software groups of data with increasingly difficulty in sample recognition and classification:

- Group 1 – full mixture: AISI 304 steel, aluminum 6082, copper and Teflon. This group is the most heterogeneous and simple to analyze, as the analyzed materials are very different one from another, with large differences in both number of peaks, intensities and peak broadening; the target is recognizing polymer from pure metals and alloys.
- Group 2 – Fe-based metals: AISI 304 steel, AISI 316 steel, C45E steel, Fe37 drawn, X150 steel. All these materials are iron-based alloys and have very similar compositions, that vary only for the ligands; the target is to distinguish the different steel grades.
- Group 3: nylon 6, high-density polyethylene (HDPE), pom-c and Teflon. All the analyzed materials are polymer-based. Even if the molecular structures are very different one from another, three of these samples (HDPE, pom-c and Teflon) have the fundamental resonant frequency in a range of 300Hz, with a noisy spectrum and broader peaks, if compared to the other analyzed materials; the target is to distinguish the polymers.

Before proceeding with the analysis, the only pre-processing that was carried out on the data was a normalization to reduce scale effects that could affect the analysis, as described on RootProf documentation [42]. As well, the range of frequencies that were analyzed spans from 0 Hz to 8000 Hz. No other pre-treatments were used in order to analyze the data with a blind approach, without using prior knowledge on the systems. However, along with the interpretation of the results, detailed suggestions on how to better analyze the results are given. The results of the analysis for the first group of samples can be observed in Fig. 5. The scree plot (Fig. 5(a)) shows that the first three principal components, the ones that the software considers reliable, explain the 62% of the total variance of the system. The explained variance in this situation can be increased by reducing the range of analysis and the variables in which no signals are present. Three PCs were selected by the algorithm also because the software automatically recognized four groups of samples very different one from the other. In this situation, PCA positions the n clusters on the vertices of an n -dimensional solid, for the representation of which $n - 1$ -dimensions are required. Therefore, in this case, the four groups are on the vertices of a tetrahedron in a three dimensional space. In Fig. 5(b) and (c) this is confirmed by the projections of the samples in the principal component space (Score plot): PC1, PC2 and PC3 can be seen as the new x,y, and z axis, and the position of the samples are the projection of a tetrahedron on the two PC1-PC2 and PC1-PC3 planes. The four clusters are highlighted by the four coloured circles on the score plot, which are calculated by the software by using a hierarchical clusterization approach (Euclidean distances with group average method). In Fig. 5(d) is reported a colour map that represents the distances between the samples. The four different clusters are highlighted by the four blue squares along the diagonal of the matrix, which represent that the ten elements of each cluster have very little distances one from another, while they have greater distances between one group and another. This approach can be without effort extended to the analysis of thousand of data and, in a quality control vision, the result automatically analyzed to identify sample not conform to a defined standard and/or outliers. In Fig. 6 the results of the analysis performed on Group 2 and 3 are reported. Results of the analysis of Group 3 are very similar to the previous ones, with a

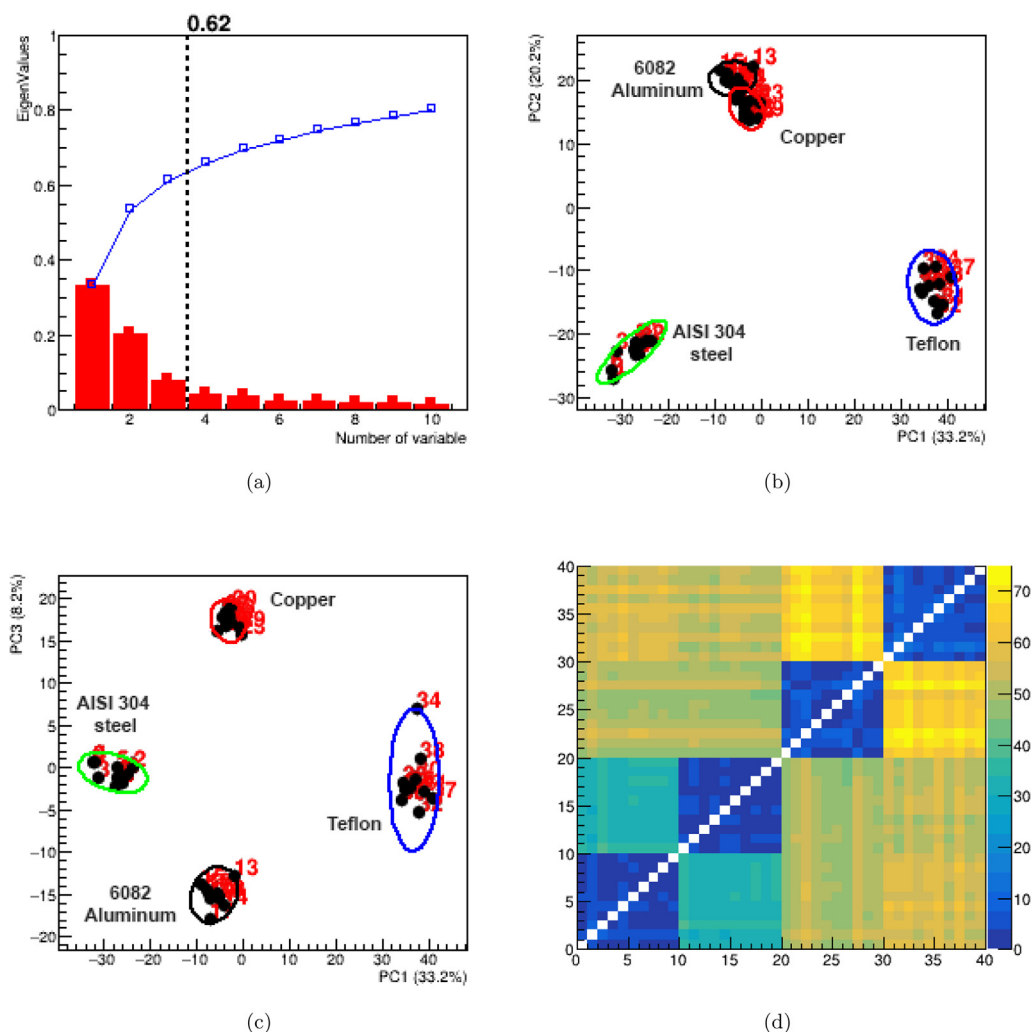


Fig. 5. Results of the qualitative analysis of the data group 1. (a): scree plot. (b): score plot of PC1 vs PC2. (c): score plot of PC1 vs PC3. (d): coloured map as representation of the distance matrix used for clusterization.

number of significant principal components which is equal to the number of samples minus one for a total explained variance of 54%. In the score plot of Fig. 6(a), samples 10–19 and samples 20–29 seem superimposed because of the projection of the data in a two-dimensional spaces, but by looking at Fig. 6(b), the two sample groups are well separated. The analysis on Group 3 samples, despite the noisy spectra recorded on the polymeric samples (showing broad peaks) gave satisfactory results, and the samples can be distinguished one from another, as shown in Fig. 6(d). The cumulative explained variance of the significant principal components is 48%. This value is rather smaller than previous three groups and is due to the noisy data. However, it must be noted that the raw data were analyzed and the explained variance (and thus the amount of extracted information, can be increased by pre-processing spectral data using pre-treatments and variable selection, commonly used in chemometrics.

- The IETeasy data collection is non-destructive, reproducible and accurate, making it suited for mechanical properties analysis. As shown at the beginning of this section, the coefficient of variation is very low ($\approx 0.001\%$ on metals and $\approx 1\%$ on polymers), demonstrating that the approach is excellent for the measurement of the properties of the materials and their classification.
- The PCA-cluster analysis, used for the first time on IET data, demonstrated that the instrument is suitable for in situ and ex situ qualitative analyses. The instrument, combined with any software for multivariate analysis is capable to recognize and classify different kinds of materials, spanning from hard materials such as alloys to soft ones such as polymers. many other materials, also of biological origin can be analyzed with the same approach.

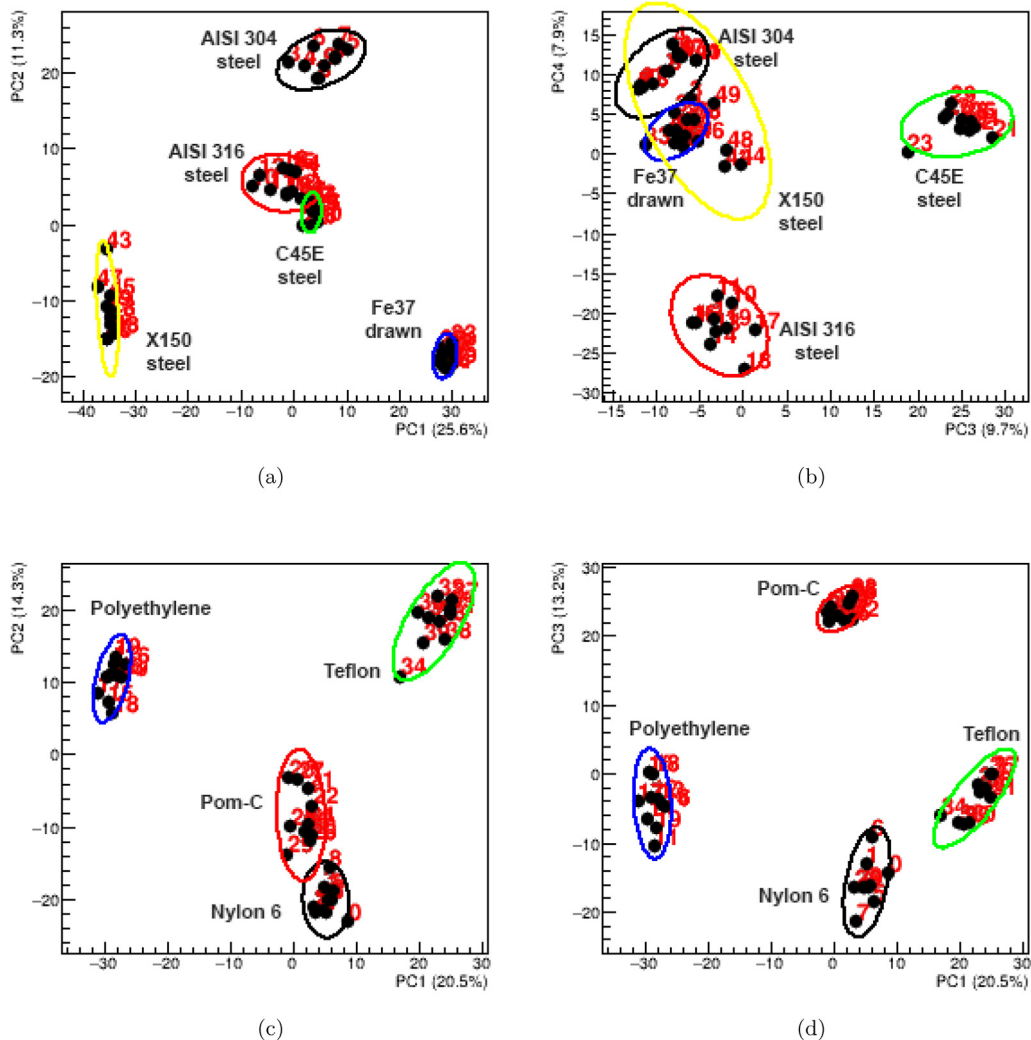


Fig. 6. Results of the qualitative analysis of the data Groups 2 (top) and 3 (bottom). (a): Score plot PC1-PC2. (b): Score plot PC3-PC4. (c): Score plot PC1-PC2. (d): Score plot PC1-PC3.

- Ellipses in score plots of Figs. 5 and 6 can be used to identify anomalies, as samples falling inside the ellipses are identified as “conform” and samples not included are “doubt” or “defective”. This approach can be further investigated as a perspective of the present work, in the quality control philosophy of detecting problems (i.e. all samples not falling within the ellipses of Figs. 5 and 6) and thus reducing scraps.
- Data collected by IETeasy can be used for classification purposes even with different methods, that can be based on Linear Discriminant Analysis which uses the single frequencies or exploiting a machine learning approach that takes as an input the whole sound profiles or the frequency spectra of the samples.
- The power of multivariate approach of PCA applied to the whole spectra analysis, demonstrated in the present paper, can be winning when handling hundreds to thousands (or even more) data file, a typical situation in quality control procedures; moreover PCA allows obtaining more information with a reduced uncertainty with respect to traditional OVAT approaches, in a much shorter time, and eventually also in an automatic way; samples not conform to a defined standard can be easily (an also automatically) identified.

Authors’ contribution

The instrument in Figure 1 was assembled by NM during his bachelor thesis under the guidance of ML. A second instrument was built by ML. Data collection was performed by NM and ML. Multivariate analysis was carried out by NM and ML. All the authors participated to data interpretation, edited the manuscript and approved its final version.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <https://doi.org/10.1016/j.ohx.2021.e00231>.

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Born in Borgosesia (VC, Italy) on February 14th, 1997. Obtained his degree in materials science in December 2020 with the experimental thesis entitled “Complete self-construction of a microcomputer-based instrument for the study of materials through impulse analysis”, under the supervision of prof. Enrico Boccaleri, prof. Marco Milanesio and mr. Mattia Lopresti at the University of Eastern Piedmont.



Born in Alessandria (AL, Italy) on January 31st, 1973. Obtained his degree in chemistry “cum laude” in 1996 with the experimental thesis entitled “Solid-gas Reactivity of organometallic complexes of transition metals”, under the supervision of prof. Roberto Gobetto and Prof. Enrico Sappa. In March 2001 he defends his PhD thesis in Chemical Sciences at the University of Turin, Department of Inorganic, Physical and Materials Chemistry. The thesis, titled “Correlation spectroscopic properties-structure: vibrational patterns applied to polyatomic systems” was written under the supervision of prof. P.L. Stanghellini. Associate professor at the University of Eastern Piedmont. Co-author of 58 articles in international scientific journals, 1 chapter in scientific volumes, 2 European patents, one patent and one Italian national patent application. Supervisor of 32 bachelor and master theses and three Ph.D. theses.



Born in Savigliano (CN, Italy) on November 25th, 1971. Degree in Chemistry in 1996 and Ph.D. in Chemistry by the University of Torino in 2001. Since October 2001 researcher and since August 2017 Associate professor in Physical Chemistry at the University of Piemonte Orientale. Recipient of the “Young Italian Crystallographer” award in 2003 and member of the teaching staff several AIC schools and organizers or chair of various international crystallographic events. Coordinator of several research projects and of a long term project (code CH-2234) at the ESRF (Grenoble, France) to develop a new instrumental setup to carry out simultaneous X-ray Diffraction and Raman experiments at non-ambient conditions. Invited lecturer at several national and international congresses. Author of more than 90 original publications on international journals, more than 50 communications to international and national congresses as main author, and 3 ITA and one EU patents. My main research interests are the study of the molecular and crystal structure of chemical compounds and materials, together with the characterization of their physical-chemical properties. Single-Crystal and Powder X-ray Diffraction, using both conventional and synchrotron radiation sources, also combined with Raman and/ or UV-vis spectroscopy, are the employed experimental techniques, also at in situ non-ambient conditions. In the last years the development of new method of data analysis in crystallography have been carried out, exploiting Principal Component Analysis for the analysis of large in situ X-ray diffraction datasets. The studied compounds are in the fields of additives for polymers and inorganic binders, of porous and layered systems for gas/molecules adsorption, transport, degradation or sequestration and of phototactic materials for photovoltaic applications.



Born in Galliate (NO, Italy) on March 17th, 1991. Obtained his bachelor's degree in materials science in 2014 and his master's degree in chemistry in 2017. From 2018 Ph.D. student in chemistry under the supervision of professor Milanesio at the University of Eastern Piedmont (Italy). His research concerns the integration of multivariate statistical analysis in the crystallographic area, especially for the quantitative analysis of powder mixtures. His Ph.D. topic is the theoretical and experimental development of lightweight X-Ray shielding materials. His work also regards coating development for ultralight magnesium alloys for everyday purposes and the analysis of plant polyphenolic extracts as adjuvants for wines and winemaking products. The red thread that connects each of these areas of interest is the passion for both crystallography and chemometric techniques.