Catas study on the composition of pencil leads

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he determination of the hardness of applied coatings using the pencil method is a procedure frequently used in different industrial sectors. In this regard, there are various standards that report the description of this method such as UNI 10782, ASTM D 3363 and ISO 15184. The principle of all these methods is the "scratching" of the coating under test using pencils of different hardness and expressing the result as the value of the hardest lead that does not scratch it. As there is currently no clear indication on the meaning of the hardness of the pencils and on its traceability to scientifically measurable parameters, this study proposes to analytically verify the composition of



the leads in order to establish a criterion for their differentiation and acceptability by the laboratories that perform this type of test.

The lead composition

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Pencil leads are mainly made up of graphite and clay to which other components such as resins, waxes and cellulose are added. The literature reports that the relationship between clay and graphite represents the fundamental parameter for the realization of leads with different hardness. The hardness, depending to the lead composition, is associated the writing effect being progressively darker as the hardness of the mine decreases. The leads are produced by initially mixing the individual finely ground components to form a sort of paste which is followed by an extrusion process that defines their geometry. The lead is then subject to a hot sintering process that produces a solid structure which is finally immersed in a wax or oil bath to fill the internal voids resulting from the sintering process. At this point the lead is ready and suitable to be used for writing and drawing both as such and after inclusion in a wooden casing (pencil).

Hardness coding

Pencil manufacturers normally use a coding system based on letters and numbers to define the hardness of a lead. There are three letters: H (Hard), F (Fine) and B (Black). The numbers, on the other hand, express the "grade" associated with the individual letters for which the 3H pencil is harder than the 2H while the 3B is softer than the 2B as it produces a darker sign (black). This system allows to define a wide hardness range ranging from 9H to 9B according to the following scale: 9H 8H 7H 6H 5H 4H 3H 2H H F HB B 2B 3B 4B 5B 6B 7B 8B 9B. As evident, in the hardness scale there are two intermediates leads represented by the HB (Hard/Black) and by the F which has been defined as "Fine" to express its evidently appreciated trait for many uses. There is neither an absolute reference for defining the hardness of pencils nor a standardized test and control method. A committee of experts from various global pencil manufacturers, has been trying for several years to develop a repeatable and reproducible standard test method within ISO, for determining the hardness of pencils, but without reaching any agreement. The hardness of the pencils is therefore still defined by each manufacturer on the basis of their own experiences, being verified through practical writing tests carried out by expert technicians of each manufacturer. These writing tests are also performed on non-standard substrates



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according to the individual procedures adopted.

The study carried out by Catas

The initial research sought to identify a simple and practical system for checking the composition of the leads used at Catas for the hardness test of coatings. On the basis of literature data and similar experiences carried out by the laboratory on composite materials (for example plastics) it was decided to use thermogravimetric analysis which allows to quantify the various components present by differentiating, in particular, the organic ones from the mineral substances.

In this way it was considered possible to discriminate at least the graphite from the clay, thus identifying the relationship between these two components considered fundamental for the characterization of the hardness of pencil leads from what is reported in the literature. The thermogravimetric analyses were then carried out with the following conditions:

- Instrument used: TGA 7 Perkin-Elmer
- Temperature program: from 25 to 900 $^\circ$ C
- Heating rate: 5°C/min
- Final step: 1 hour at 900 ° C
- Gas: N_2 up to 600°C and air from 600 to 900°C
- Sample weight: about 15 mg

The tests were conducted on individual samples considering the time length of the test (about 4 hours), reserving the repeatability of the analytical data for a possible subsequent investigation.

The results

The analyzes were carried out on the pencils in use at the Catas surfaces section (only for the lot examined). Six pencils were selected which normally correspond to the hardness of the coatings tested by the laboratory.

	Additives	Graphite	Clay
	(%)	(%)	(%)
2H	10,1	49,3	40,6
Н	10,6	52,2	37,2
F	9,7	53,8	36,5
HB	12	59,3	28,7
В	11,7	61,9	26,4
2B	12,6	62,2	25,2

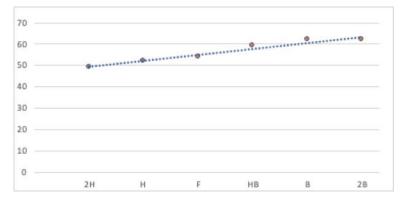
Table 1. Relative composition of the leads analyzed



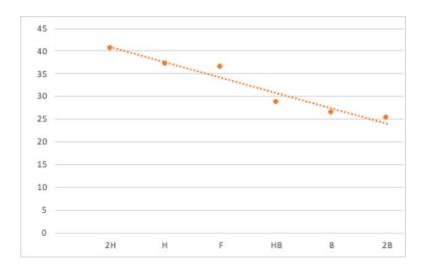
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The processing of the thermograms obtained made it possible to obtain the following results expressed in terms of percentage composition.

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Graph 2. Percentage of clay as a function of the hardness of the leads

Comments and conclusions

The examination of the thermoanalytical curves shows a significant differentiation in the percentages of graphite and clay among the various leads with a clear indication of the trend in the clay/graphite ratio (higher ratio for higher hardness) in accordance with the initial hypothesis. The apparent "linearity" found deserves further investigation as it may not be significant as there is no objective evidence that the hardness of the various leads correspond to a regular variation in their basic composition. However, the data found are still encouraging as they suggest the possibility of defining an analytical criterion for the control of the composition of the leads to which their hardness value is obviously associated.



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Future developments

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To make the proposed analytical method usable, it is believed that it is necessary to plan the following additional activities:

- performing repeated tests on the same batch and on different batches of pencils from the same manufacturer;
- performing similar tests on pencils from several manufacturers.

In principle, at the end of these further investigations, it will be possible to establish whether:



a. the identified method can be used as an absolute system for verifying the composition of the leads independently of the producer in the event that, obviously, all producers use the same "recipes" for the production of leads;

b. the identified method can be used as a system for verifying the constancy of the composition of the leads of each individual producer in the event that each producer has its own specific recipe for the production of leads;

c. the method, while valid, cannot be used in the event that the recipes are already variable within each individual manufacturer, as the hardness value is a purely empirical data obtained from empirical writing tests.

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