Determining the concentration of diamond powder deposited on a textile yarn: a multitechnique approach

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Aims

The development of an analytical method to determine the amount of diamond powder deposited on a textile yarn arises from the need to certify the concentration of diamond itself in textile fabric. Due to the fact that diamond textile yarn had to be used to produce luxury and high fashion manufactures, the certification was an absolute need in the marketing of such products that are proposed with a remarkable price to the final users.
The production line of the diamond wire was developed by a company of Prato (Tuscany, Italy) textile area. In figure 1 a very simplified scheme of production is reported. A special apparatus drives the wire through a container where a mix of viscous resin, diamond powder and glitter is present. The mixture is stirred during the whole process. The thread, once soaked in the viscous mixture and enriched with diamond powder, is dried and then rolled ready to be used.

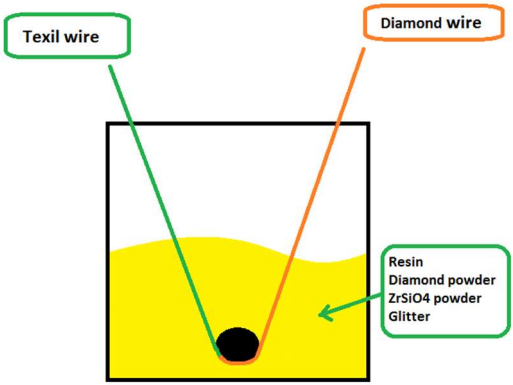

Figure1. Schematical representation of the apparatus used to produce the diamond-enriched textile yarn

The analytical issues could be pointed out as follow:
1) verification of the presence of the diamond powder on the final fabric and as consequence on the textile yarn,
2) determination of the linear concentration of diamond on the textile yarn,
3) verification of the homogeneity of the product sample, *i.e.* the concentration should be constant throughout the whole process production.
Diamond is pure carbon and, as a consequence, practically indistinguishable, through the normal analytical techniques, from cotton and resin that are both made up as well mostly by

carbon. The determination of the concentration of diamond in these conditions has been represented as a real challenge for all the analytical laboratories that had been involved.

Method and results

CRIST (Crystallography Center - Università di Firenze) is an academic facility with a wide range of available X-ray instrumentations, most of them dedicated to X-ray diffraction analysis. Then, the first idea was to verify the presence of diamond in the final fabric through an X-ray diffraction analysis. In fact, diamond, thanks to its crystalline structure, well diffracts X-ray.
The fabric was analyzed with a Bruker D8 diffractometer, configured with Bragg-Brentano geometry, using Cu Kα radiation, the diffractometer was equipped with a Euler cradle in order to allow the positioning and orientation of the specimen (*Figure 2*)

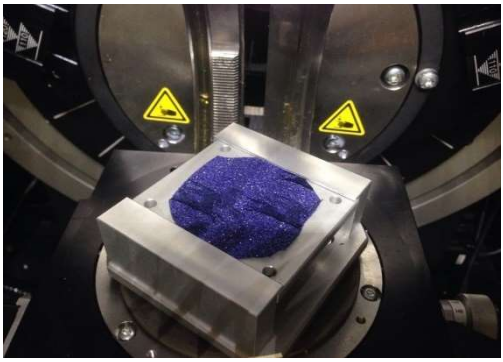

Figure 2

The XRPD (X-ray Powder Diffraction) pattern in *figure 3* shows the characteristic peaks of the diamond at 43.92° in the 2Θ range 43-45° angle Thus XRPD analysis[1] was useful to easily solve the first analytical question confirming the presence of diamond on the finished fabric.

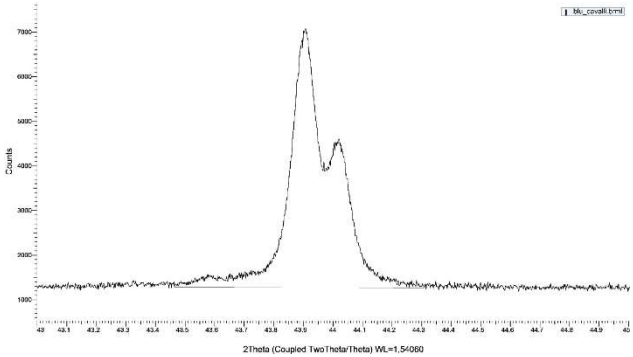

Figure 3. Fabric XRPD pattern

Unfortunately, the XRPD technique is not easily suitable for an absolute quantitative determination. Thus, to give an answer to the remaining analytical questions, it was necessary to follow a different approach.
The diamond is pure carbon so its mass attenuation coefficient (MAC) is too similar to those of resin and cotton substrate MAC, therefore, μ-Tomography (μ-CT) analysis did not seem, at first glance, helpful for our analytical purposes.
In fact, as evidenced in figure 4, where the 3D rendering of the analyzed textile yarn is reported, a first μ–CT analysis confirmed the impossibility to distinguish the diamond from the wire (the spots highlighted in green are due to the glitter grains).

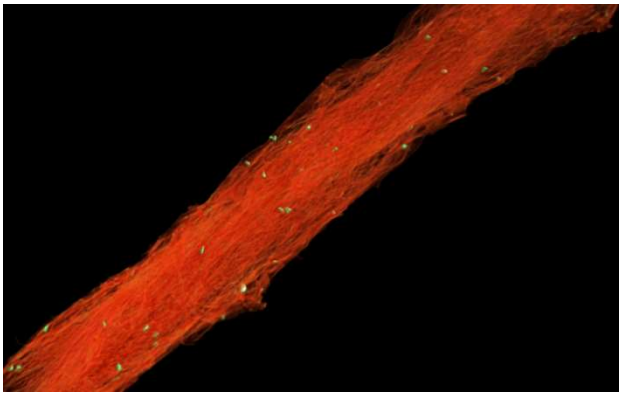

Figure 4

Then in order to solve the analytical problem we started to think in a different way, and we consider the only other component (well visible by using the μ-CT device) present in the fabric yarn, *i.e.* the glitter. The glitter describes a wide assortment of small fragments mainly consisting in the copolymers, small sheets of aluminum, titanium dioxide, iron oxide, bismuth oxychloride and other oxides and/or metals, painted with iridescent colors capable to reflect the light in the visible spectrum.
The glitter has the function to shine the light. In fact, the diamond powder is grey as it absorbs light, does not have a high optical dispersion index and, as a consequence, it has not the brilliance of the cut stone.
The XRPD pattern of the diamond powder, figure 5, shows the high peak of the diamond itself (red), the $Zr_{0.87}Y_{0.214}O_{1.7}$ Zirconium Yttrium Oxide (blue) and some other not identified impurities.

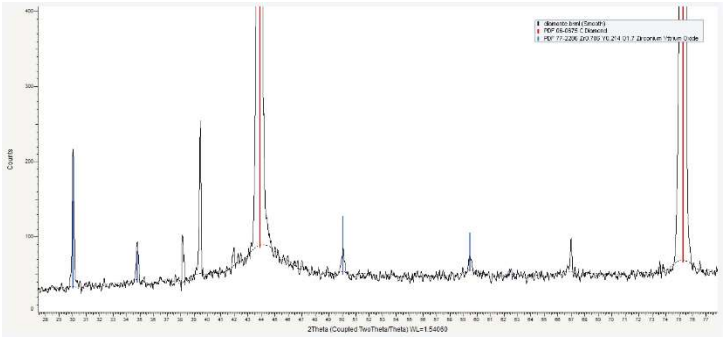

Figure 5

The analysis of the diamond powder by wavelength dispersion X-ray fluorescence spectroscopy (WDXRF) has confirmed the presence of Zr, Y and some other elements as reported in the following table:

Element/Compound	Result	Detection limit	Element Line
C	99.9%		
P2O5	0.0751%	0.00079	P-Kα
CaO	0.0203%	0.00055	Ca-Kα
SiO2	0.0114%	0.00095	Si-Kα
ZrO2	0.0059%	0.00022	Zr-Kα
Fe2O3	0.0025%	0.00050	Fe-Kα
Y2O3	0.0019%	0.00021	Y-Kα
MgO	0.0016%	0.00147	Mg- Kα
Al2O3	0.0013%	0.00060	Al-Kα
CuO	0.0004%	0.00030	Cu-Kα

Table 1

These preliminary results suggested us to add a small weighted amount of an inorganic compound containing zirconium to the diamond powder to get a strong x-ray absorption contrast with the carbonaceous matrix of the diamond yarn. Zirconia was excluded due to its radioactivity, and zircon, $ZrSiO_4$, was chosen as perfect candidate. The zircon sand was sieved with 120µm first and then 80µm mesh, this step selected grains with a known size range. The sieved sand was mixed to the diamond powder in known proportions, then the powder mixture was kneaded with the resin. Progressively the proportion of silicate was increased taking care that the standard quality level of the textile yarn was preserved. The mixture used had ZrSiO4 / diamond ratio equal to 1: 4. Samples were withdrawn in different times during the production process and they were analyzed with a µ-CT, Skyscan 1172[2]. Four meters of yarn rolled on a plastic support was analyzed in each scan. The zircon grains are clearly visible as shown in figure 6.

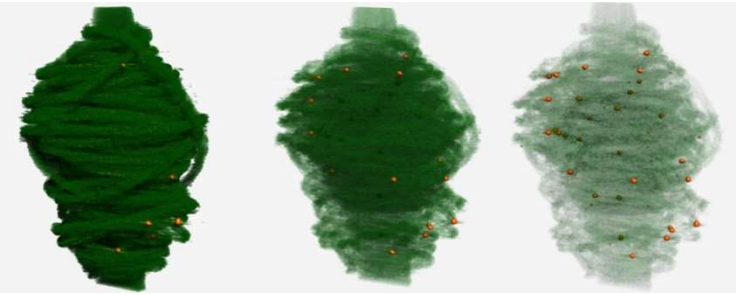

Figure 6

The measuring conditions were selected to better define the size of the zirconium silicate grains and to exclude any glitter contribution.

Filter	Cu+Al
Resolution	19,5 µm
Rotation step	0,44°
Voltage (kV) Current (mA)	100kV 100mA
Hounsfield Unit range	0-4000Hu

The left part of figure 8 is a 3D rendering of a portion of a roll of yarn acquired in the same condition reported in table 2 but without primary filters, and the relative reconstruction carried out by selecting a proper HU range to highlight either the wire, the particles of ZrSiO4 (cyno circle) and the glitter (red circle). The right section of figure 8 shows the same sample acquired and reconstructed with the conditions reported in the table 2, only the ZrSiO4 grain are displayed.

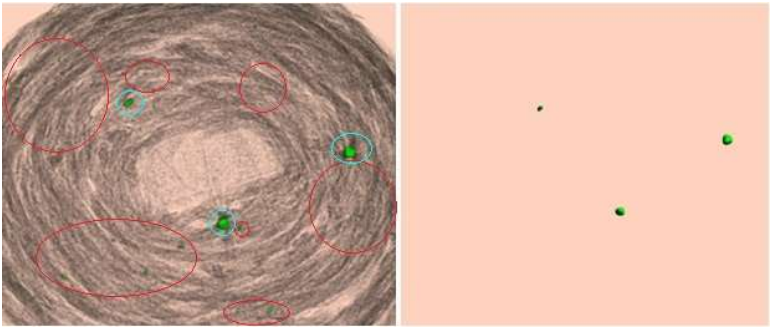

Figure 8

With the knowledge of the range grain size of the sand it was possible to adjust the correct threshold to use for the determination of the total grain volume. As it shown by the thickness

distribution, most of the identified $ZrSiO_4$ particles are within the range between 80 and 120µm:

Structure thickness distribution			
Range (mm)	Mid-range (mm)	Volume (mm^3)	Percent volume in range (%)
0.020 - <0.060	0.040	0.00071	1.6251
0.060 - <0.100	0.080	0.01655	37.7031
0.100 - <0.140	0.120	0.02663	60.6717
Standard deviation of structure thickness: 0.02mm			

The concentration is calculated just applying the formula:
$[(d_{ZrSiO4} \times V_{ZrSiO4})/M_{sample}]x4= M_{diamond}$ gr/Kg
Where:
d_{ZrSiO4} is the density of $ZrSiO_4$ sand
V_{ZrSiO4} is the total volume of the grain sand measured from µ-CT analysis
M_{sample} is the total mass of the yarn used for µ-CT analysis
$M_{diamond}$ is the concentration expressed in gr of diamond for Kg of yarn

Analysis of the production batch 1402N1S is reported. Four samples were analyzed: Test1 from the beginning of the production process, Test2 and Test3 from two intermediate times, and Test4 at the end of the process. The expected concentration value (expressed in gr of diamond over Kg of yarn) was 7.5gr. Table 3 summarized the results from tomography analysis:

Sample	Sample weight	gr. of diamond over Kg. yarn
Test1	105mg	6.54-8.20gr.
Test2	72mg	4.65-5.50gr.
Test3	103mg	7.16-8.70gr.
Test4	103mg	6.73-8.52gr.

The concentration is reported within a minimum and a maximum value that correspond to the two limits of threshold used in the binarization step during the particle volume determination. The measurement accuracy for Test1, Test3, Test4 is acceptable for the final purpose of the analysis and the average value is almost constant throughout the process production. Test2 shows a lower concentration value respect to the other three results: it's important to notice that in this case just 72mg of yarn was used for the analysis, such smaller amount of sample was not statistical representative of the ZrSiO4 distribution.
The residue amalgam mix (resin silicate of zirconium and diamond in the container) at the end of the production process was also analyzed via XRPD. The semi quantitative analysis calculated from the diffractogram revealed exactly a relative concentration of 20% for ZrSiO4 (red) and 80% for diamond (blue). That is the same composition before the beginning of the process, this result confirms that the ZrSiO4/diamond ratio remains 1: 4 during the whole process production.

Conclusion:

The multitechniques approach was successful in all the analytical issues. In fact, XRPD easily identified the diamond powder either in the final fabric and in the textile yarn. The concentration of zircon on the textile yarn was determinated by micro tomography technique. The homogeneity of the product sample was verified by micro tomography technique and supported by the XRPD analysis on the residue resin. The combination of results allowed us to evaluate the diamond concentration on the yarn.

Acknowledgement:

We thank the Product Manager **Dr. Alessia Beconcini** (Industrie Bitossi, Sovigliana, Vinci) that supplied us the zircon sand.

References:

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