Analysis of Asbestos in Bulk Materials by X-Ray Diffraction: Influence of Grinding Methods on the Result

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Abstract

The grinding method for sample preparation affects the results of X-ray diffraction analysis of minerals, mainly the soft and inelastic components of raw materials and synthetic matrices. This fact is determined by a “disturbance” of crystal order in the surface layer of particles and is more pronounced when a fine comminution is performed and when a high shearing stress is applied in grinding. This effect is remarkable on asbestos minerals.

Subsequent to some information on the development of the science of “mechanochemistry”, this paper reports the results of several tests on single, binary and polygenic materials containing chrysotile and crocidolite under different grinding conditions (type of mill and grinding time). The aim of the research is to give suggestions about a sample preparation methodology for XRD quantitative determination of asbestos in bulk materials, suitable for obtaining more accurate results.

1. Introduction

According to the policy of the European Community in environment protection, it is currently necessary to know the rate of asbestos minerals in raw materials and synthetic matrices used for different applications and in the building industry. These materials are always polygenic and often very complex, as in the case of serpentinite rocks, cement-asbestos aggregates, insulating plates and waste materials containing chrysotile, amosite and crocidolite. The analytical problem has no easy solution and often requires different technical approaches because the grinding methods can alter the analytical response, which depends on the crystalline lattice of solids.

This is the case of X-ray diffraction (XRD) analysis, the results of which are connected to the mechanical, thermal and chemical stress the samples have been subjected to.

The present work deals with some historical backgrounds on the subject, in connection with the new field of the mechano-chemical sciences; it will also report on some tests on single, binary and polygenic materials with the aim of discussing the methods of grinding the samples for XRD quantitative determination of asbestos in coherent materials.

2. XRD analysis on very fine powders

X-ray analysis of dust and fine powders was used for a long time for the study of occupational diseases which are connected with the nature of airborne dusts (e.g. silicosis). For this type of analysis, the new surfaces arising from the fracture and the fine comminution of solids, as well as the layer immediately underneath represent an anomaly; their properties are accountable for the conditions that have caused the breakage.

These remarks have been considered for a long time in scientific literature with reference to several physical and chemical characteristics; Scherrer (1918) [1] mentioned the “distorted crystal aggregates” and defined the effects of both the decrease in the crystallite size and the lattice distortion on the XRD response. The degradation of crystalline perfection was described by Stokes (1944) [2] and Bacon (1952) [3], while later on, some mineralogical changes connected to the milling action were reported, e.g. transformation of calcite into aragonite [4].

Several hypotheses on the nature of the superficial layer of particles were suggested, mainly with regard to quartz. A “disturbance” in the crystalline lattice, not negligible in terms of mass for particles less than 0,5 μm in diameter, was supposed to be corresponding to a superficial “amorphous” or “amorphyzed”, highly soluble layer (in borate or sodium hydroxyde dilute solutions), “which blends smoothly into the less soluble core” [5] [6].

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A layer was estimated to be 20-30 nm thick (as resulting from specific surface determinations by permeability method, or by solubility tests), or 30-50 nm thick (from densimetric measurements, with reference to the volumic mass of quartz and amorphous silica).

In several tests [7] the well-known phenomenon of XRD line broadening was not evident, except for the region of back reflection where the diffractometric response (determined by photometric analysis on film) was lowered to 66% (pure quartz, ground under 2 μm) and to 58% (only classes of less than 1 μm). The reduction in peak intensity was observed to be much less when grinding was applied to foundry sands (quartz and minor quantities of soft accessories): in the narrow size range (0.5-1 μm), it was inferred that the high soluble surface layer is about 30 nm thick, based on the mass determination of amorphous silica removed from the grain surface by dilute hydrofluoric acid.

Some contrasting hypotheses were derived from the observation of an increased solubility also on the surface of vitreous silica; some authors [8] did not accept the theory of the amorphized layer on particles and suggested the hypothesis of a "disorder" in crystallite orientation. Some others [9] assumed that three different layers are present on the new particle surface: a mono-layer of hydrated silica, then a disturbed crystalline layer slowly soluble in dilute hydrofluoric acid and finally an ordered core. The disturbed layer produces the line broadening which is more evident on grain sizes of less than 2 micrometers.

It was subsequently proved that the nature and thickness of structural anomalies on the particle surface were due to grinding methods [10] by ascertaining that an increasing grinding time results in both a decrease of the number of observed spectra and in an increase of the background intensity. In contrast, when quartz was ground by hand milling in the presence of acetone, both a strong broadening of the X-ray reflection lines and a small decrease in their integrated intensity were observed. The particles did not lose their angular contour; this led one to believe that — when dry grinding was adopted — a region of the particle close to the surface momentarily melts and becomes amorphous on cooling.

A confirmation of the importance of the nature of solids (particularly as regards the deformability of weak minerals) on the XRD response was obtained by the author in 1963 [11]. In dry grinding 5 types of asbestos [12], a great reduction in height of the lines and even a disappearance of peaks was observed when a strong shearing stress was applied by protruding milling action by means of agate hand mortar and pestle for 30 minutes. In contrast, the use of a wet blade-blender or ultrasonic grinder prevented the re-agglomeration and sintering of solid microparticles and satisfactorily maintained the original diffractometric response. Figure 1 [13] clearly represents the evolution of XRD response of a sample of crocidolite as run-of-mine (A), ground using a small hammer-mill (B), a wet blade-blender (C) and a dry agate mortar (D) for 15 min (1) and 30 min (2), respectively.

During the 2nd European Symposium of Comminution (Amsterdam, 1966), several effects of "amorphization" in relation to grinding time (with a vibration mill) were discussed [14] with reference to quartz, galena, rutile, magnetite, and cryolite. Quartz, in particular, revealed a reduction of the (101) peak from 3200 to 100 imp/s after 200 hours of comminution, the outcome differing according to the milling charge (corundum, iron) and the presence of additives. A ball mill with a porcelain pot [15] was also used to grind some zinc oxides (up to 384 hours!): the characteristics of grain-size, density, colour and specific surface of the products were determined and some phenomena of re-agglomeration identified and discussed, in relation to crystallite size and lattice distortion, as verified by the roentgenographic method.

3. Structural modifications in solids induced by mechanical action and "Mechano-Chemistry"

The term "mechano-chemistry", coined by Ostwald [16], is currently employed to characterize the complex physical and chemical phenomena caused by the mechanical treatment of solids [17]. For a long time, however, these properties have been utilized in ore beneficiation, for flotation and selective flocculation, to control the inter-phase behaviour of solids, liquids and air.

Some meetings on mineral processing dealt with many problems of mechano-chemistry, for example, regarding the industrial grinding of quartz, hematite, boemite, hydragillite, bauxite, titanomagnetite, anatase and kaolin [18]. In some cases, grinding time reached 280 hours; the importance of ore hardness and composition as regards XRD response was ascertained; e.g. when treating some bauxites, a milling duration of "only" 20 hours removed all diffraction traces.
Mineral engineers have more recently analysed the whole phenomenon of comminution from an energetic point of view, in terms of specific surface, energy activation and relaxation and on the basis of thermodynamic and crystallographic theories [19].

The differences between soft and hard compounds with respect to grinding behaviour, amorphization and chemical transformation following comminution were presented in the field of chemical and materials engineering as a new tool for the production of advanced materials [20].

Langer et al. (1978) investigated the consequences of the mechanical action on fibres of chrysotile asbestos [21]. They first examined the structural modifications induced by stress and fracture on both Si-O and Mg-O interlayer bonding and brucite layer configuration. The decrease of "fibre crystallinity" produces some variations in the physico-chemical properties of finely ground products. Changes in XRD peak areas, IR and ESR spectra and SAED patterns were also analysed: unground and simply classified short fibres or fibres obtained by a "gentle" size reduction or cutting action have a much more relevant crystalline response than products of a "vigorous" ball-mill grinding, where impaction, compressive and shearing stresses are associated. Further
implications of these changes may also involve biological responses such as hemolytic activity, organic free radical absorption and carcinogenic potency of short fibres.

Ultra-fine grinding is nowadays investigated as a cause of deep modifications in the surface layer of solid phase, which may determine changes in bonds, aptitude to selective ion adsorption and zeta and hemolytic potentials [22]. Some recent researches concern:
– the consequences of ultrasonic treatment on the properties and the physico-chemical behaviour of particle surfaces, similar to a mechanical action [23];
– interaction between solid and fluid phases after a mechano-chemical treatment of quartz and asbestos [24];
– surface active sites (atoms, ions, dangling bonds) and molecular modifications consequent to comminution [25];
– chemical functionalities, present on the extremities of mineral fibres, and their increase by mechanical rupture [26].

Some interesting examples of changes in the structure of solids and in the related reactivity consequent to fine comminution were given in a recent paper in this magazine [27]: “mechanical activation” may produce mechano-luminescence (e.g. tribo-luminescence), emission of radiations (fracto-emission), transformation of molecular composition (as in mullite, produced by high-energy grinding of kaolin).

4. Processing problems in asbestos sample preparation for XRD

The sensitivity, reproducibility and accuracy of the quantitative determination of mineral components can be strongly affected by variations in the diffractometric response of the crystalline structure caused by grinding (type and time). For this reason, it was formerly suggested [28] that when examining rocks and synthetic materials containing asbestos, analysis should be performed on unground samples. The problems arising from large-sized crystals and/or preferential fibre orientation in raw samples should, of course, be carefully considered.

A XRD method of high sensitivity and accuracy for chrysotile determination was developed by the I.S.S. ("Istituto Superiore di Sanità."). Rome [29]. Later on, a wet comminution system for bulk materials as well as an appropriate analysis were tested on asbestos-cement samples [30].

A method of producing asbestos-cement comparative standards was recently proposed by the Italian Institution for Standardization (UNI) [31]; this procedure carries some risk of error due to some processing uncertainties. It was therefore deemed useful to analyse some aspects of sample comminution.

4.1. XRD analysis for asbestos

ISS suggests the use of a silver filter as the support for 0.4 mg of powdered material, obtained from a slurry previously ground with a small vibrating mill in the presence of isopropyl alcohol, and then dried and sampled.

In contrast, the UNI proposal assumes the analysis of samples of about 1 g, consisting of powder contained in normal sample-holders; actually this type of support is easier to obtain and assures better accuracy of the final result, provided a uniform volumic mass concentration is realized in the powdered sample.

For this reason, we use the latter support for this analysis, which requires the preparation of several grams of raw material.

4.2. Preparation of reference standards

The case of cement-asbestos waste products, to which the UNI proposal is directed, cannot be oversimplified by assuming that these materials are made only of asbestos and a generic “cement” powder. In fact, besides calcite and quartz (present at various rates in commercial cement products), the old and altered cementos-asbestos plates and tubes contain some products of mortar hydration, hardening, transformation and alteration which are not well represented in the raw cement: the reference standards should represent the real possible composition of the samples to be analysed.

4.3. Classification and concentration of samples for analysis

The UNI proposal for preliminary classification of the dust released from bulk samples on a nylon screen is somewhat debatable due to a possible electro-static selective separation of some components from the original sample. The initial beneficiation of all materials by means of a dilute hydrochloric acid attack seems to be restricted to instances of real necessity, resulting from the preliminary microscopic analysis: even a weak acid dissolution could in fact damage the integrity of the finest chrysotile fibres. Nevertheless, it can be useful in that it eliminates great quantities of calcite, when present in the original samples.
4.4. Grinding of samples
In its experiments, ISS used a small special wet grinding apparatus (McCrone micromilling mill), which is useful for avoiding a damaging local temperature increase. The volume of the grinding cell, therefore, is rather small as compared to the mass of sample usually required by XRD analysis. On the other hand, in the UNI standard proposal only two parameters are specified, i.e. the use of a generic hand mortar and the maximum size of the particles (63 μm): such a grain size for test standards seems to be too large and must be confirmed by suitable tests.

For all the above reasons, an attempt to provide a preliminary answer, at least to some of the following questions, seemed useful:
1) what is the XRD response for some pure minerals, in relation to the milling action?
2) when milling is carried out on binary artificial mixtures, what are the results for the two different components?
3) what is the XRD response on the single components when grinding is applied to a cement-asbestos composite containing several types of asbestos, calcite, quartz, and accessories, in addition to the setting and transformation products of cement mortar?

5. The experimental work: materials and methods
The following minerals and materials (and some of their binary associations) were tested:
1. Quartz (hydrothermal crystalline, from Quincinetto, Torino),
2. Calcite (rose marble from Candoglia, Novara),
3. Chrysotile (industrial product “5 M” from Balangero, Torino),
4. Old asbestos-cement commercial product (a corrugated plate that roofed a shed for 20 years: it contains 2 types of asbestos, calcite, quartz and accessories).

When suitable, a preliminary comminution was carried out using a small hammer pestle to reach a grain size of less than 63 μm, then an intermediate grinding of 1 g of the sample was carried out using an agate mortar with 120 dangling movements, exerting an orthogonal force of about 20 N, and final grinding was executed on single 0.3 g samples using the same manual tool, with a rotating trajectory of the pestle, exerting a shearing stress under a normal force of about 20 N for 1, 2, 4, 7, 10 min (2 r.p.s.). In some cases, grinding was prolonged up to 20 min (binary mixtures) and 40 min (chrysotile).

A small toothed disc mill and a blade laboratory grinder were also utilized to grind a sample of an asbestos-cement composite; this device can reduce the sample particles to under 0.05-0.1 mm, dependent on the operation time.

Characterization of the ground products was obtained by means of optical microscopy (phase contrast and polarized light); the specific surface was calculated from the grain-size distribution from the Rosin-Rammler plot.

The XRD analysis was performed using a Philips PW 1800 apparatus with a Cu anode tube (40 kV generator voltage, 50 mA current, 10 mm irradiated length on sample, 0.02° step size, 1 s time per step, 5...65°, 2θ angular prospecting). The sample (130 mg) was placed in a circular cell (14 mm in diameter, 0.4 mm thick), fixed by 2 drops (50 mg) of vaseline oil, 100 r.p.m. rotation speed.

The peaks were considered corresponding to the following characteristics: quartz (d = 0.246 and 0.343 nm), calcite (0.304 and 0.191 nm), chrysotile (0.736 and 0.366 nm), crocidolite (0.835 nm). The XRD response was determined by evaluating both the maximum height and integrated peak intensity.

As the aim of the experiments was mainly to verify the interference between hard and soft minerals as a consequence of the type and duration of grinding action, without any calibration for quantitative analysis, all results have a preliminary significance and must be considered only as an example of progressive selective degradation on the diffractometric response of mono- and polygenic materials.

6. Results
6.1. Monogenic powders
Several tests on quartz, calcite and chrysotile were performed to confirm and to complement the conclusions drawn from a number of old experiments [11]. Qualitative results for chrysotile confirm a progressive decrease of XRD response, which is more important for the line heights (final reduction after 10 min. grinding to 40% for 2θ = 12.2° and 34% for 2θ = 24.4°) than for the integrated intensities (to 48% and 44%, respectively).

A lesser degree of reduction was observed on calcite, where final results (lines at 2θ = 25.5 and 47.7°) were 65% and 68% of the original values and 80% and 70% in integrated intensities, respectively.

On quartz powders, there was a minor reduction limited to the final grinding phases (86% and 89%);
90% and 91% for lines and integrated intensities, with reference to peaks on \(2\theta = 20.9^\circ\) and \(26.7^\circ\), respectively.

For chrysotile, the reduction in the XRD response was minor during the first period of dry grinding, becoming higher after 5-10 min of mechanical action: the trend was asymptotic at values of 20-40% of the original state after 40 min grinding (wherein in the past all crocidolite response was found to be nearly cancelled in the same conditions).

This phenomenon also depends on the roughness of the grinding mill surfaces and is linked to a new aggregation state of particles smaller than 2-3 \(\mu\)m in size, which are compacted in aggregates that cannot easily be destructed, even by wet dispersion.

Chrysotile grinding in presence of \(H_2O_2\) has also been observed to reduce the statistical dispersion of both the integrated intensities and background; further investigations are required on this subject.

6.2. Binary mineral associations

The response of some mixtures of chrysotile (10%) with quartz or calcite, when ground using a similar procedure, has been studied. As for single minerals, the height reduction of the peaks is greater for the weak components. However, for binary mixtures the height reduction of the peaks for weak components is lower than for single minerals. In fact, it seems that large crystals of rigid and hard minerals such as quartz could protect the weak ones, preventing their micronization and a corresponding strong reduction in their XRD response.

When grinding a mixture of 90% calcite and 10% chrysotile for 20 min, the chrysotile line at \(2\theta = 24.4^\circ\) was unidentifiable, whereby the main peak (\(2\theta = 12.2^\circ\)) is just visible, together with a great line broadening (integrated intensity 45% of the primary one, for a simple homogenization of pure components).

6.3. Sample of cement-asbestos corrugated plate

The fibrous components of this tile-building material turned out to be amphibole and serpentine asbestos in nearly equal rates (abt. 30% in all).

The changes in XRD response for the main lines of chrysotile (\(2\theta = 12.2^\circ\) and 24.4\(^\circ\)), crocidolite (\(2\theta = 10.65^\circ\)), calcite (\(2\theta = 25.5^\circ\)) and quartz (\(2\theta = 26.7^\circ\)) were examined.

The evolution of peak heights and integrated intensities was similar to the previous examples (Figs. 2 and 3). Attention was also paid to the specific surface of the powders: the graph (Fig. 2) was plotted on the basis of the grain-size distribution, as determined by optical microscopy on calcite, quartz and cement crystals, disregarding asbestos fibres. The letters indicate the XRD response of the products obtained by the toothed disc (D), the manual pestle for intermediate grinding (P), and the blade grinder operating for 2 and 5 min., respectively (B1 and B2).

![Graph](https://example.com/graph.png)

Fig. 2 Evolution of the XRD response (I: height of peaks) against milling time (t) for the main lines of chrysotile: \(C_1 = (002)\), \(C_2 = (004)\), amphibole asbestos chrocidolite (A), calcite (CA), quartz (QZ). S is the specific surface of powders; B1 and B2, D and P the treatment by a blade mill (2 and 5 min), a toothed disc grinder and an agate mortar (with a compression action only), respectively.

![Graph](https://example.com/graph2.png)

Fig. 3 Evolution of the XRD response (I: integrated intensities, in conventional units) against milling time (t) (Symbols as in Fig. 2).
With reference to the grinding product obtained using the hand pestle after 1 min of impact (P), the two main lines of chrysotile (and the corresponding integrated intensities) denote a concordant but not proportional reduction.

As in previous examples, the attenuation of XRD response for calcite was somewhat smaller than for asbestos and was very small for quartz. A maximum value for diffracted intensities was found for short grinding times (for instance, by exerting pressure and impact using a manual pestle for 1 min and by obtaining a grain-size distribution smaller than 0.04-0.05 mm, with a 30...50 m²/kg specific surface).

In terms of specific surface, the XRD response of asbestos minerals is represented by some linear regressions (see Fig. 4, for the principal peaks of chrysotile, (002) and (004) reflex, and for the main line of crocidolite, and Fig. 5 for the respective integrated intensities). These regressions are represented by the following relations:

\[ I_i = 112 - 0.048S; \quad I_{i1} = 21.8 - 0.0103S; \]

\[ I_2 = 60 - 0.032S; \quad I_2 = 13.4 - 0.007S; \]

\[ I_3 = 252 - 0.136S; \quad I_3 = 19.2 - 0.0104S \]

where \( I \) and \( I_i \) are the heights and integrated intensities (as counts per second and conventional units, respectively), 1, 2 and 3 are the two main lines of chrysotile and the first reflex of crocidolite, \( S \) the specific surface, as previously defined, in m²/kg. The quoted relations \( I(S) \) are valid in the ranges \( S = 500...1,200 \text{ m}^2/\text{kg}; \) the \( I_i(S) \) in the greater range 150...1,200 m²/kg.

It is clear to see that crocidolite is more sensitive to grinding than chrysotile, so that its response decreases with comminution is greater; consequently, crocidolite was no longer detectable in a significant way after 10 min of milling action, even if its original percentage by mass was 10-15%. The results of dry comminution using suitable laboratory grinders are comparable with those obtained by a hand mortar and pestle, operating for 1-1.5 min.

7. Final remarks

Due to the great number of physical parameters involved, the results of this research cannot be generalized. They may be valid to assess the selective degradation of mineral and synthetic materials, according to the particular method of comminution.

The XRD response is strongly influenced by the "crystallite disorder", dependent on elastic and mechanical properties of the mixtures and aggregates, and reaches the maximum value for soft, ductile and inelastic components. However, it may vary under the same grinding conditions in accordance with the composition of the materials and particularly with the simultaneous presence of both hard and soft constituents.

Great attention should be paid to the method of getting samples for roentgenographic analysis and calibration standards, as the components may interfere with each other and may imply further complications.
7.1 Grain-size of samples for analysis
When operating with cement-asbestos materials, it is possible to obtain reproducible and accurate results with powders ground below 0.04-0.05 mm.

7.2 Grinding time
Milling duration should be related to the grindability and original sizes of materials, as well as to their crystalline texture; in general, the grinding time should be kept at the shortest level necessary to obtain the above reported sizes: 1-3 min are often enough, for both hand mortars and mechanical disc or blade grinders.

7.3 Grinding devices
Dry grinding by means of agate mortars is acceptable, provided that the shearing stress on samples and relied attrition effects are reduced to a minimum. If preliminary and intermediate screening are employed, overgrinding and its negative effects on the XRD response can be avoided; multi-stage grinding and screening are useful, provided that all sample particles are sufficiently reduced to pass the pre-determined mesh size. The wet grinding method (in an alcoholic medium, as with the McCrone micronizer) should be confined to scientific research on very small samples, to be analysed after dispersion and filtration on suitable membranes.

7.4 Sample supports
When the holder is a simple disc cell, it is possible to reduce the sample mass to a small fraction of 1 gram [32], weakly fixed with vaseline oil when necessary. However, a constant volumic mass concentration in the powder layer should be ensured; this can easily be obtained with the normal pressed tablets.

Filters (cellulose or silver) can be confined to the mono-layer analysis for very small quantities of powder or dust.

7.5 Calibration standards
The more difficult step for XRD analysis on multiphase powders is to obtain a suitable standard. Calibration tests should be performed on the materials to be investigated. This is because the variability of diffraction angles reported for some international standards and by different authors is sometimes large: for instance, a variation interval of 0.65° 2θ (for the main peak) and of 0.9° 2θ (for the second) was quoted for kaolin, which can show some interference with asbestos minerals [33]. Similarly, the relative values of integrated intensities can be quite different for different types of the same asbestos (i.e. in the case of ortho- and clino-chrysotile and for some fibrous amphiboles).

Furthermore, a great flexibility in choosing the reference lines should be adopted by taking into account the possible interference of the associated components in commercial and natural materials; calibration curves should therefore be prepared on the basis of all identifiable and significant peaks.

7.6 Sensitivity and accuracy of analytical results
On the basis of the above remarks, it does not always seem plausible to achieve a detection limit as low as 1%, as stated by many scientists: a sensitivity of 2-4% was often found for polygenic substances. Finally, the accuracy of XRD quantitative asbestos determination can be acceptable when—according to the bulk material composition—the standard deviation of the experimental values is less than 20%. One should note that for more complex associations of asbestiferous materials, only a multiphase analytical method (always based on a careful microscopical analysis, with both polarized light and phase contrast technique) can reach good sensitivity and accuracy.

This analysis should be extended to all intermediate and final grinding products, to obtain complementary information on the nature of the components and the grain-size distribution.

8. Conclusions
1) The quantitative determination of asbestos minerals, and in general of soft and inelastic minerals, with the X-ray diffraction (XRD) analysis method is influenced quite strongly by the grinding of the sample. This fact is due to modifications of the crystalline structure in the surface layer of solids, which results in a reduction of the peak heights in the XRD response.

2) This reduction of the peak heights in the XRD response largely depends on the type of grinding method and on the grinding time, and reaches the maximum value for a grinding action based mainly upon shearing and attrition stresses.

3) As a consequence of the above, particular care must be taken in selecting and applying standard procedures for the preparation of the samples and of the calibration standards to be subjected to the XRD analysis.

4) For the preparation of XRD samples from asbes-
to the calibration standards, which must accurately reproduce the quality and the size distribution of the material to be analysed.

6) In order to overcome several uncertainties of the analyses, some advantages are obtained by combining a preliminary microscopical examination (polarized light and/or phase contrast) with the usual XRD procedure. In this way, more information about both the composition and size distribution of the powders may be obtained.

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Nomenclature

d Reticular distance \( \text{nm} \) \( (10^{-9} \text{m}) \)

I Diffracted intensity \( i/s \) (impulses/s)

I\text{r} Integrated intensity convent. units (dimensionless)

r.p.m. Revolutions per minute \( \text{min}^{-1} \)

r.p.s. Revolutions per second \( \text{s}^{-1} \)

S Specific surface \( \text{m}^2/\text{kg} \)

t Grinding duration \( \text{min} \)

2\( \theta \) Diffraction angle degree \( ^\circ \)

ISS Istituto Superiore di Sanità (Italian Institution for Health)

UNI Unificazione Nazionale Italiana (Italian Institution for Standardization)

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