# Assessment of quartz materials crystallinity by x-ray diffraction

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**Abstract.** The estimated degree of crystallinity of natural and synthetic grown quartz and quartzite by calculating the x-ray diffraction patterns. It is shown that the index of crystallinity of natural quartzite varies widely, reflecting the different degree of their transformation. The highest values of the index of crystallinity are characterized natural and synthetic single crystals of quartz.

## 1. Introduction

Despite of the fact there were numerous studies of structural and physical properties of quartz, the interest in quartz content is not waned. This is because of the wide use of products made from natural quartz, the usage of synthetic quartz crystals with the desired properties and natural polycrystalline quartzite as a source of high-pure quartz raw materials, as well as to study the conditions of their mineralization and recrystalization.

To evaluate the degree of conversion and perfection of crystalline quartz [1] it was offered to use the "crystallinity index", measured by the parameters of the x-ray diffraction spectra. In the paper [2] it was proposed to use the method of infrared spectroscopy for evaluation the degree of chalcedonies conversion.

The evaluation of the degree of crystallinity can be detected with the most pure forms of natural quartz material – quartzite [3...5], which can be used in high technologies. This paper describes the study of crystallinity index of natural, synthetic quartz crystals and natural polycrystalline silica quartzite that was held with the XRD method.

## 2. Research methodology

To determine the degree of quartz material conversion, samples of fine-grained quartzite of the Antonov group of deposits [6] (the Deposit "Sopka-248" and "Belokamensky"), quartzite deposits (Borel-Saridag, Buryatia) [7], granular quartz (a field is Malokutulak, Yakutia), natural smoky quartz (Polar Urals), natural Brazilian optical quartz were studied.

In addition, the samples of artificially grown quartz from different conditions were studied for comparison with obtained results. These are: highly pure quartz synthesized in the laboratory of the Institute of Mineralogy SB RAS, Novosibirsk, Russia (samples provided by L. G. Shcherbakova), samples of synthetic quartz with an admixture of aluminum and polychrome zoned crystal of quartz with an admixture of two or trivalent iron [8] grown in the all-Union research Institute of synthesis of mineral raw materials (samples provided by V. Balakirev).

Furthermore, samples of fused quartz and of technical quartz glass was also studied with XRD method.

For this research fine-pounded samples were taken, which were compressed into a "pill". The measurements were performed on the diffractometer X"Pert PRO. Radiographs were taken with increments of about 0.02 in the range 5...70 deg., with the rotation of 30 rpm and a shutter speed of 0.1 sec at a point.

After filming on the diffractometer X Pert PRO x-ray samples under Cu Kα–radiation with a minimum speed and higher sensitivity, x-ray spectra was received. They cover the whole range of possible angles.
If necessary, radiographs X-axis can be derived interplanar distances, calculated in accordance to the software in the form of labels on the Y – axis the intensity of diffracted lines. The results of the registration of x-ray diffraction spectra were processed by a special program with data output in digital form [9].
The index of crystallinity Kci was carried out according to the method of Murata and Norman [1] who proposed to use a multi-blade block parameters (quintuplets) peak in the region of 67°and 69° on the x-ray diffraction pattern. All the obtained radiographs of the estimated maximum quintuplets peak corresponds to the value of 2θ = 67,7797° ± 0, 0003, unlike proposed in [1], the values of 2θ = 67,74°.

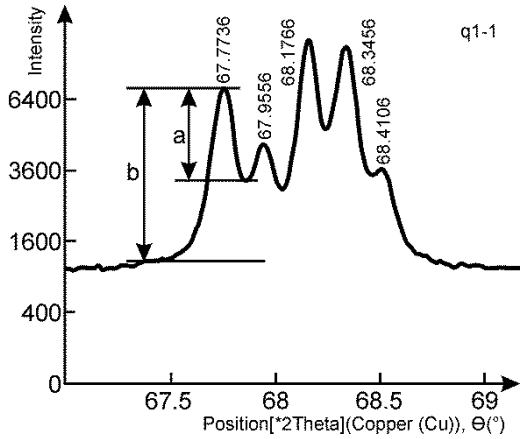


Fig. 1. An explanation of the calculation of the crystallinity index by the method of Murata and Norman [1] in the parameters produce multiple peak in the region of 67°and 69° on the x-ray diffraction pattern

To calculate the index of crystallinity according to the formula K=10 F a/b proposed in work [1] the experimental values of the intensity of the peak at 2θ = 67,77° (Fig. 1) was used: a/b – ratio of the intensity of the peak at 2θ = 67,77° to the intensity of the background; F is the scaling factor used for assessment of the crystallinity index of K = 10 reference spectra crystal optical quartz (Madagascar). In the calculations, the scaling factor was taken equal to the unit.

## 3. Experimental results and discussion

In diffraction patterns of fused quartz the multiple peak at 67°and 69° is not observed indicating the absence of crystalline phases (table), that is also confirmed by the results of the studies by infrared spectroscopy.

**Table.** The index of quartz crystallinity of different materials, calculated by the method of x-ray diffraction

| Sample | Crystallinity index Kci |
|---|---|
| Fused quartzite / quartz | 0 |
| Quartzite (deposit Sopka-248) | 2.88…6.78 |
| Quartzite (deposit Belokamensky) | 4.65…6.71 |
| Superquarzities (deposit Bural-Sardag) | 6.66…7.34 |
| Granular quartz (deposit Malokutulak, Yakutia) | 7.84…8.24 |
| Natural smoky quartz | 7.33…7.48 |

| | |
|---|---|
| Synthetic quartz (Institute of synthesis of mineral raw materials) | 7.32...7.84 |
| Synthetic quartz (Institute of Mineralogy SB RAS) | 7.20...7.32 |
| Brazilian optical quartz | 7.51…7.55 |
| Quartz glass | 8.48…8.57 |

Crystalline quartzite of "Sopka-248 and Belokamensky" deposits are characterized with a wide range of calculated values of crystallinity index. The cleanest fine-grained varieties of quartzite have low values of K ≈ 2.88.

There is a change quartzite chemical composition and color with depth, as well as from the Central portions of the ore body to the periphery; the degree of their crystallinity increases up to values of 6.67…5.38. In local areas, especially in areas of high fragmentation, initially chemically pure quartzites under the influence of supergene processes decrease their quality characteristics and the degree of crystallinity increases up to 6.7.

Increasement in the degree of crystallinity of quartzite from Antonov group of deposits is connected to the irregular processes of metamorphism of siliceous biogenic sediments, resulting in the crystallization of amorphous silica and a crystalline phase α-quartz, which is also confirmed by infrared spectroscopy [10].

Quartzites with superquality from field Bural-Sardag are comprised larger grains with inclusions of porphyritic elongated, subparallel orientation, which indicates their formation (recrystallization) under prolongated dynamic stress [7]. In the thin cross sections the regenerated fragments of quartz were found. High values of the index of crystallinity (7.34…6.66) indicate a high degree of their transformation.

Granular quartz of Malokutulak deposit came under the processes of dissolution due to the passing of the intergranular boundaries of the mineral-forming solutions and the subsequent dynamic recrystallization, which led to formation of polygonal structure of quartz with sinuous grain boundaries, characterized by high K values in the range of 7.84…8.24 comparable to the estimates of the crystalline natural quartz.

Crystalline samples (natural and synthetic) quartz have the highest values of the index of crystallinity. Minimal variation in Kci shows the sustained processes of crystallization and formation of a committed and well-ordered crystals.

Technical quartz glass is characterized by the absence of the far right in its structure. It consists of microcrystal grains of quartz, and in the spectra of x-ray diffraction reflects the presence of crystallinity (see table).

## 4. Conclusion

Obtained by x-ray diffraction values of the index of crystallinity, reflect the degree of conversion of quartz raw materials: the highest values characterized by granular and crystalline (natural and synthetic) quartz. Despite of the fact that values calculated by the proposed method, the index of crystallinity of quartzite are very relative, it can be used for comparative evaluation of the degree of metamorphism of the quartzites (in combination with the methods of infrared spectroscopy) within the same field.

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