Expert for Failure Analysis and Mineralogical Assessment of Building and Inorganic Materials

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Expertise No. 7042729-Summary

Summary
based on the expertise No. 7042729

Optoelectronic, Physical-Chemical and Phase-Analytical Investigations on Neuburg Siliceous Earth by means of Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX), Cathodoluminescence in the SEM (SEM / CL), X-Ray Diffraction (XRD), Infrared Spectroscopy (IR) and Thermo Analysis (DTA / TG)

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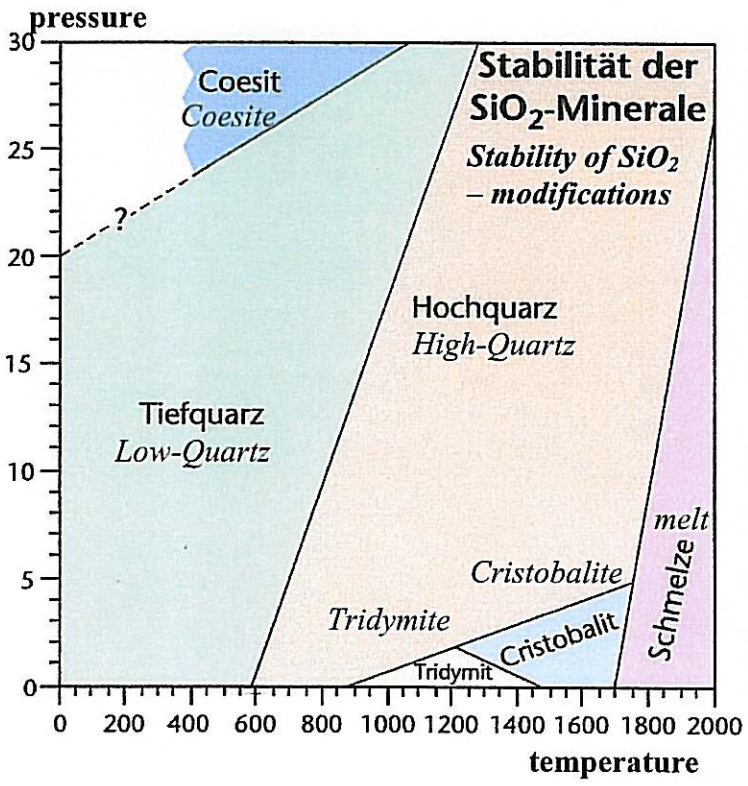
General Preliminary Remarks on SiO_2 - Modifications

At different conditions of temperature and pressure SiO_2 forms a series of polymorphic modifications, of which the most important are with respect to the mineralogical, crystallographic denomination: Low – (alpha-) – Quartz, High – (beta-) – Quartz, Tridymite, Cristobalite, Coesite and Stishovite. Further on there still exists a series of rare (Lechatelierite, Melanophlogite, Keatite …) and synthetically produced SiO_2 –modifications.

The following illustration (picture 1) schematically shows the currently accepted pressure / temperature relationship of stable SiO_2 – modifications.



At ambient pressure the following modifications exist according to RÖSLER (1991):

- **(Low-) Quartz up to 573°C**
- **High-Quartz from 573°C up to ~870°C**
- **Tridymite from 573°C up to ~1470°C**
- **Cristobalite**

Picture 1:
Stability of SiO_2 – modifications in the pressure – temperature diagram (Stosch 2003).

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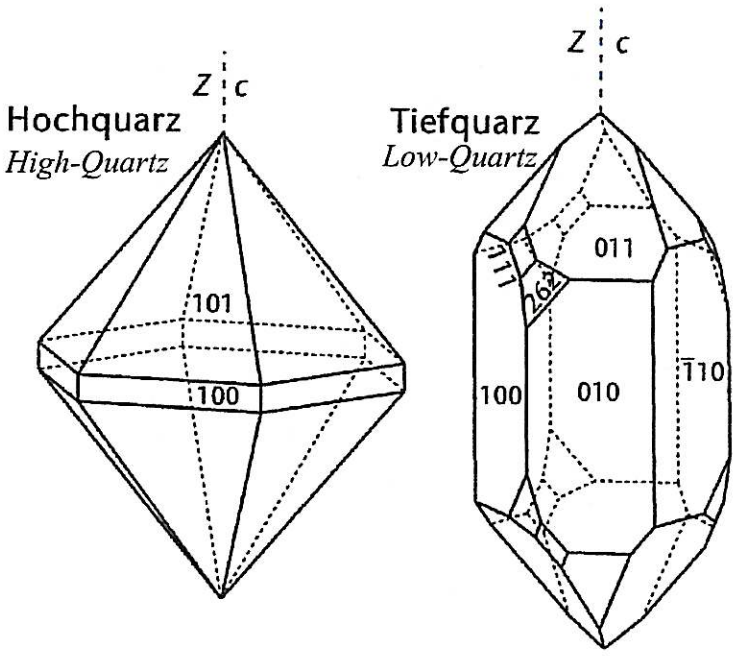
- The transformation Low - / High – Quartz is enantiotropy (transferable into each other), or easily reversible, proceeds very rapidly in a narrow range of temperature and shows 1% change of volume. This transformation does not need any de-bonding and new formation of Si – O bindings.
- Except High-Quartz all crystalline SiO_2 – phases and the melt (so-called undercooled melt) can be generated and maintained meta stable at ambient condition.
- At a temperature of higher than 573°C originally separated High-Quartz **always** exists as Low-Quartz, whereas the morphology of the primary High-Quartz may be conserved after the spontaneous transformation into Low-Quartz.



As native Low-Quartz appears as well formed crystallites in different shapes, the crystallites of naturally formed High-Quartz mostly develop hexagonal di-pyramids. The faces of the hexagonal di-pyramids may be shortened significantly or are left totally.

Picture 2:
Typical morphology of High-Quartz and Low-Quartz (Stosch 2003).

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As the use of the labeling alpha – and beta – Quartz or Low- and High – Quartz is not standardized within international publications, discrepancies may occur. In former German-speaking publications the Low – Quartz was mentioned as beta – Quartz, the High – Quartz as alpha – Quartz – in contrast to English-speaking publications, where the Low – Quartz was called beta – Quartz and the High – Quartz was called alpha – Quartz. In the following the more logical denominations using Greek figures as alpha for low temperature followed by beta and so on for higher temperatures became more and more accepted.

General Preliminary Remarks: Neuburg Siliceous Earth

The classical Neuburg Siliceous Earth (also labeled as Sillitin and Sillikolloid) is a native mixture of corpuscular silica (silica acid) and lamellar Kaolinite. Both mineral phases build up a distinctive conglomerate. On the basis of the silica's fine grain size, of its round shaped grain and its naturally aged surface a unique structure of particles is generated.

According to HOFFMANN MINERAL GMBH & Co. KG the investigated specimens Sillitin V85 and Sillitin Z86 reveal the following mineral phase constituents amongst others:

Sillitin V85: 79% silica, 15% Kaolinite, 6% Accessory Minerals
Sillitin Z86: 68% silica, 25% Kaolinite, 7% Accessory Minerals

(Data in weight %)

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CONCLUSION

Expertise statement: Unique features of Neuburg Siliceous Earth

- **The thermo-analytically investigated specimens Sillitin V85 and Sillitin Z86 do not show a Quartz transformation behavior at 573°C, compared with other investigated SiO_2 – specimens.**

- **The investigated specimens Sillitin V85 and Sillitin Z86 definitely contain a combination of amorphous and crypto - crystalline SiO_2 – modifications.**

- **An originally at temperatures of > 573°C precipitated High- (beta-) SiO_2 - modification always exists as Low- (alpha-) SiO_2 - modification at ambient temperatures, whereas the morphology of the primary High- (beta-) SiO_2 - modification may be conserved after the spontaneous transformation into the Low- (alpha-) SiO_2 - modification. Hence the Low- (alpha-) SiO_2 - modification exists significantly as a pseudomorphosis to the High- (beta-) SiO_2 - modification in the Neuburg Siliceous Earth.**

- **The morphological proof of the High- (beta-) SiO_2 - modification definitively succeeded. Well formed, hexagonal di-pyramids could be identified by scanning electron microcopy.**

- **The crystallites of the pseudomorphosis of the High- (beta-) SiO_2 - modification are affixed among each other with an amorphous SiO_2 - matrix, partially coated opal - like or melted into a mineral entity. Their surfaces are aged and show an amorphous, opal - like structure.**

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Project	Neuburg Siliceous Earth	Contact	Herr Dr. Christian Seeger		
Customer	Hoffmann Mineral GmbH & Co. KG	Date	January 21, 2008	Seite	6

- **Cathodoluminescence effects of the SiO_2 – modifications indicate that they were formed above 573°C and rapidly cooled down.**

- **In my judgment the Neuburg Siliceous Earth – represented here by the investigated samples Sillitin V85 and Sillitin Z86 – can neither even proximately be produced synthetically nor by the mixture or ad-mixture of native components of a SiO_2 – modification with Kaolinite.**

On the basis of the scientific results and because of the high degree of disordering of the SiO_2 – modification the Neuburg Siliceous Earth can be characterized as a unique SiO_2 – modification respectively the SiO_2 – modification contained in the Neuburg Siliceous Earth may be labeled as Neuburg Silica.

I am at your disposal for further questions

Date: Neunkirchen am Sand, 21.01.2008

Dr.rer.nat. Jürgen Göske

Von der Industrie- und Handelskammer Nürnberg für Mittelfranken
Dr. rer. nat. Jürgen Göske
Schadensuntersuchung und mineralogische Beurteilung von Baustoffen und anorganischen Materialien
öffentlich bestellt und vereidigt

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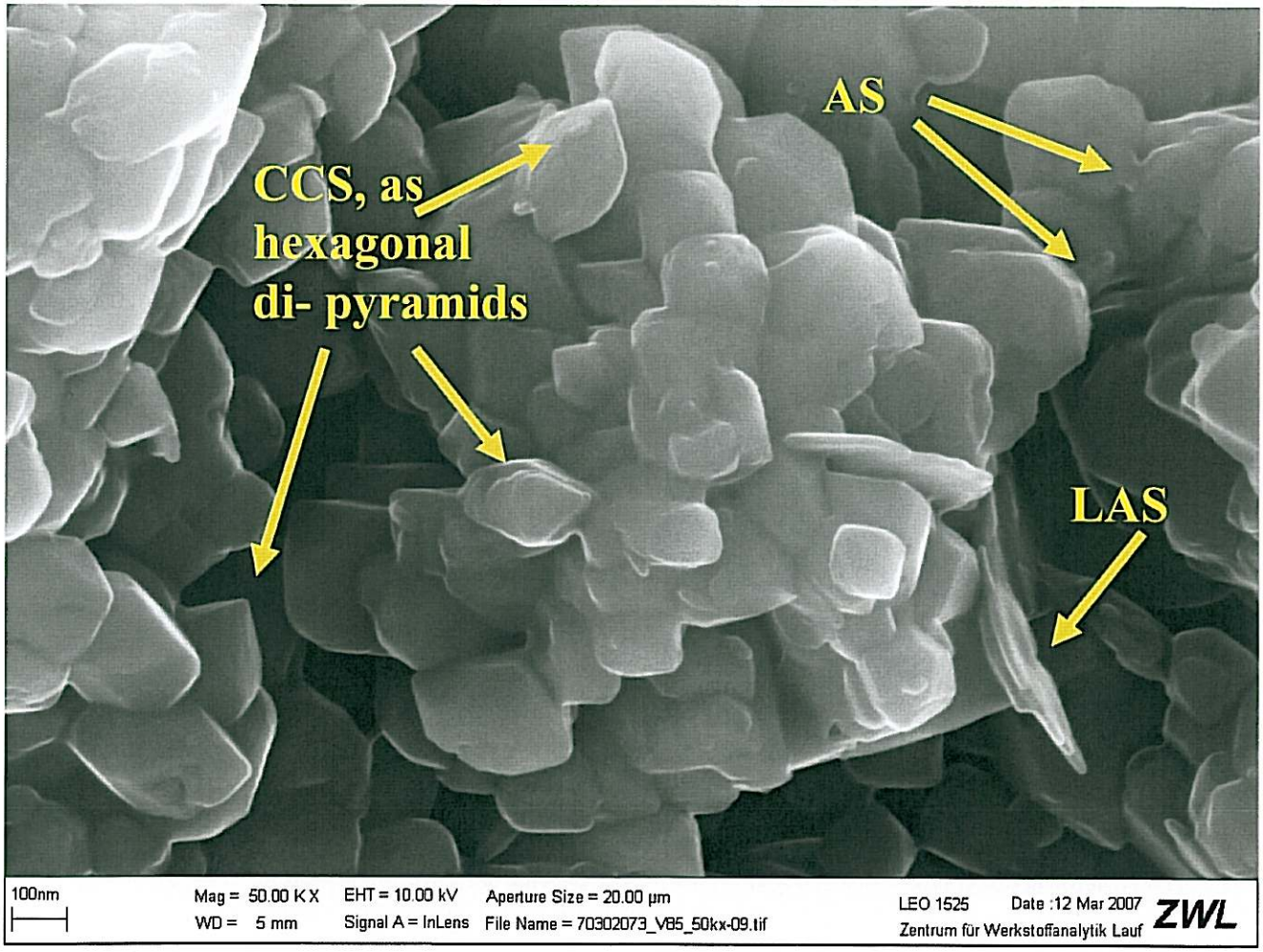
Appendix: SEM-images



SEM – image of Sillitin V85 – quarter sample I / 2007, image acquired on 12th Mar 2007. Besides crypto - crystalline (CCS) and amorphous (AS) SiO_2 – phases there are visible lamellar Alumo-Silicates (LAS). No isolated SiO_2 – crystallites could be identified; all of them are affixed and stuck together by an amorphous – like SiO_2 containing matrix, coated opal – like with the amorphous – like SiO_2 containing matrix.

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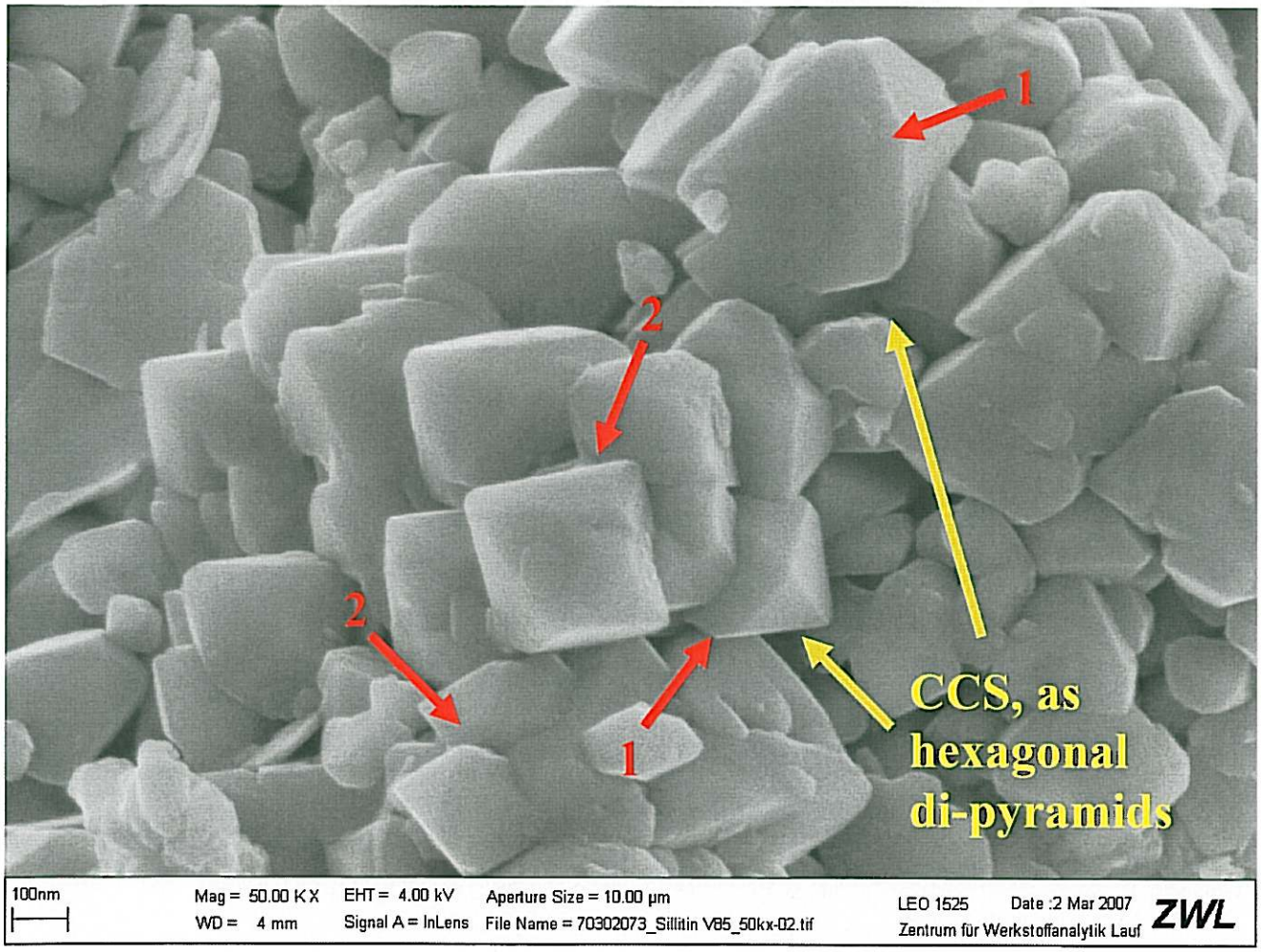

SEM – image of Sillitin V85 dated on 2nd Mar 2007. The single crypto - crystallites (CCS) of the pseudomorphosis of the High- (beta-) SiO_2 - modification are not isolated. They are all affixed and stuck together by an amorphous - like SiO_2 containing matrix, coated opal - like with the amorphous - like SiO_2 containing matrix. The faces of the hexagonal di-pyramids may be shortened significantly (1) or are left totally (2).

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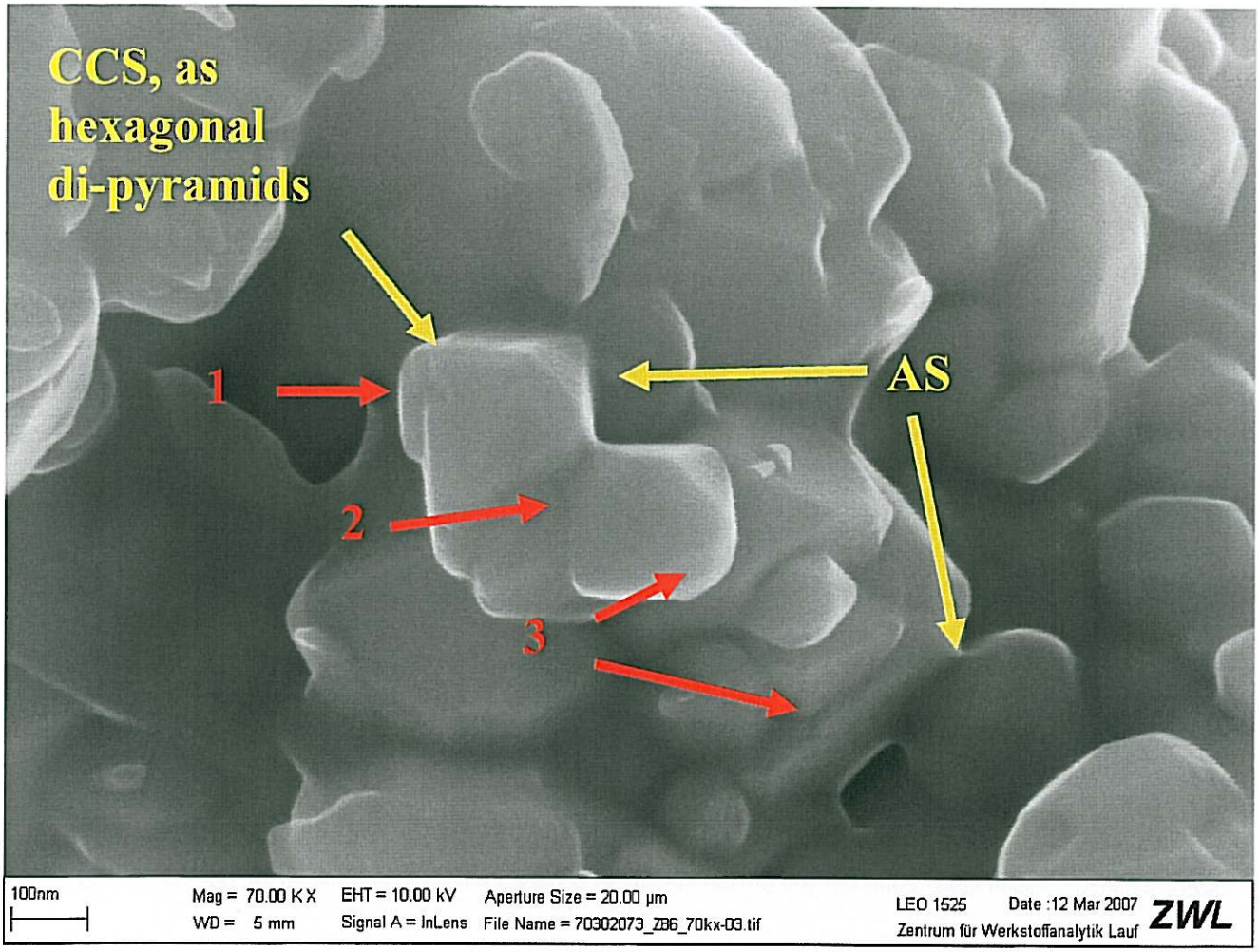

SEM – image of Sillitin Z86 dated on 2nd Mar 2007. Single crypto-crystallites (CCS) of the pseudo-morphosis of the High- (beta-) SiO_2 - modification stuck together and are affixed by an amorphous - like SiO_2 containing matrix (AS), partially coated, bonded or molten into a "mineral entity". The faces of the hexagonal di-pyramids may be shortened significantly (1) or are left totally (2). Single crystallites are aged superficially and show amorphous, opal – like structures (3).

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