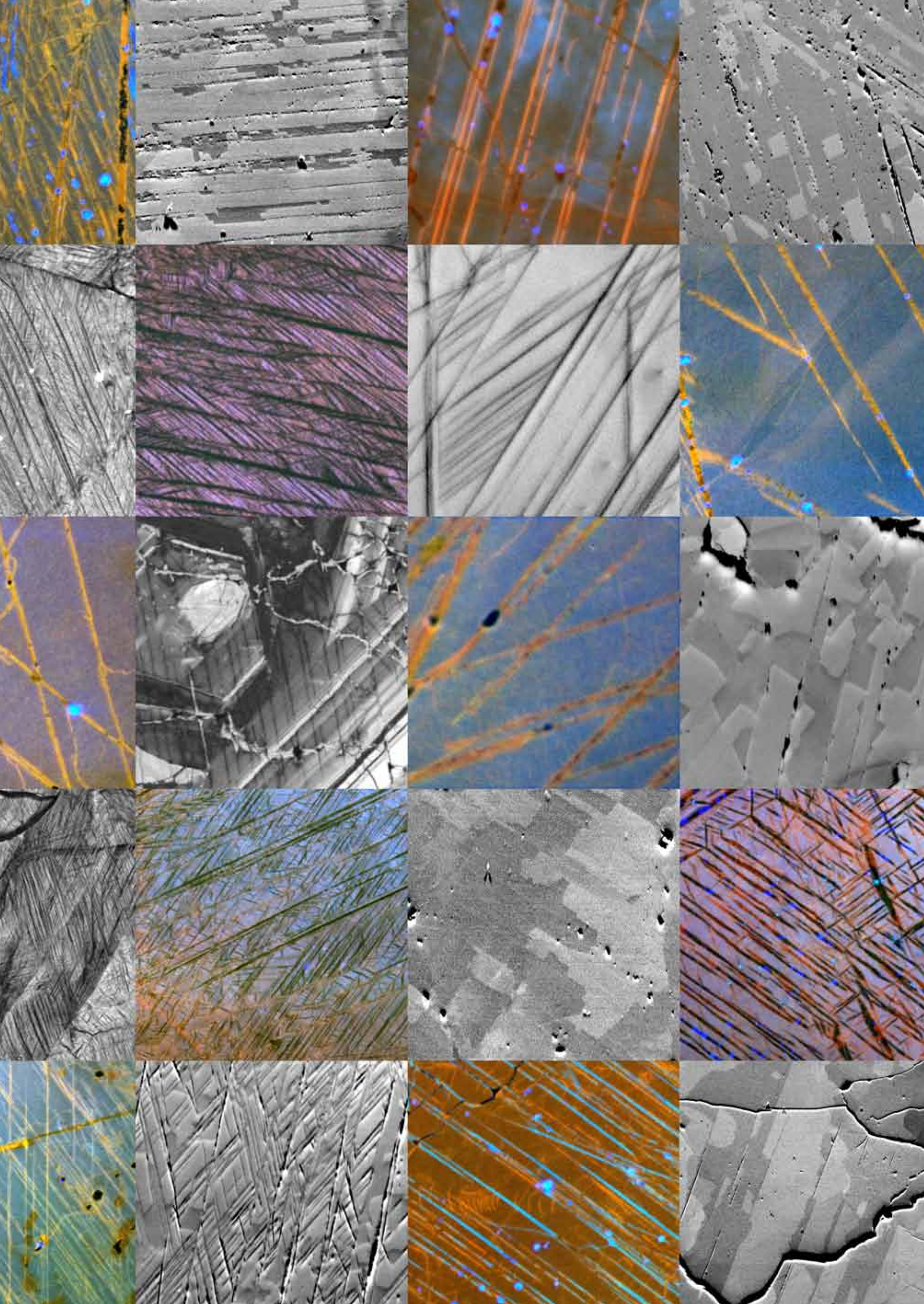

Contents

Chapter 3

No evidence for shocked quartz at the Cretaceous-Palaeogene boundary in the Geulhemmerberg section, south-east Netherlands

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The Cretaceous-Palaeogene (K-Pg) boundary section in the Geulhemmerberg caves, south-east Netherlands, might be one of the most complete and continuous shallow marine K-Pg sections known. The globally observed impact ejecta layer at the boundary is missing, indicating a hiatus at the boundary, but traces of elevated iridium content and the presence of organic biomarkers near the boundary suggest that the hiatus is limited. Material from the impact layer may be preserved in burrows extending from the boundary into the latest Cretaceous sediments. To check this hypothesis, burrows extending from the K-Pg boundary level in this section were checked for shocked quartz by light microscopy and scanning electron microscope-cathodoluminescence (SEM-CL) imaging. If the hiatus at the boundary is indeed negligible, remains of the Chicxulub ejecta layer that is found worldwide at this level should be present, such as shocked quartz grains. However, no planar deformation features in quartz were found, but instead all planar or sub-planar microstructures were tectonic deformation lamellae or healed microfractures. Therefore the continuity of the section cannot be confirmed. The successful application of SEM-CL imaging confirms the potential of this technique for the study of shocked quartz.

3.1 Introduction

An ejecta layer containing impact material from the Chicxulub crater in Mexico has been recognised at the Cretaceous-Palaeogene (K-Pg) boundary (66 Ma) worldwide and is often used to identify the exact level of the K-Pg boundary. The impact layer contains impact spherules, Ni-rich spinel, high iridium (Ir) content, nanodiamonds and shocked minerals such as quartz, feldspars and zircon (Alvarez et al., 1980; Bohor et al., 1987; Carlisle and Braman, 1991; Morgan et al., 2006; Smit and Hertogen, 1980; Smit and Kyte, 1984). Of these, shocked quartz grains are the most abundant and easily detected feature, which is furthermore resistant to post-deposition alteration. Shocked quartz grains display one or (usually) more sets of planar deformation features (PDFs), which are diagnostic for meteorite impact and straightforward to detect in a standard petrographic microscope (Grieve et al., 1996; Stöffler and Langenhorst, 1994). However, other (sub-)planar microstructures in quartz are frequently misidentified as PDFs, especially tectonic deformation lamellae (sometimes mentioned as Böhm lamellae or metamorphic deformation lamellae) (Ernstson and Fiebag, 1992; Ernstson et al., 1985; Langenhorst and Deutsch, 1996; Langenhorst et al., 2005; Retallack et al., 1998). Reliable distinction between the two types of microstructure can be difficult in a light microscope and transmission electron microscope (TEM) observations are often required to prove the shock origin of planar microstructures in quartz (Grieve et al., 1996; Stöffler and Langenhorst, 1994). Recently, scanning electron microscopy (SEM) cathodoluminescence (CL) techniques were proposed as an easy and quick alternative method to identify PDFs in quartz and distinguish them from non-shock related (sub-)planar microstructures (chapter 2; Boggs et al., 2001). In this study, we used SEM-CL imaging to check the presence of shocked quartz grains at the Geulhemmerberg K-Pg boundary in the Netherlands.

3.1.1 The Geulhemmerberg Cretaceous-Palaeogene boundary section

The Geulhemmerberg K-Pg boundary section, exposed in manmade caves in the Geulhemmerberg in Limburg, the Netherlands, has been described in detail in a special issue of Netherlands Journal of Geosciences (Geologie en Mijnbouw) in 1996 (Brinkhuis and Smit, 1996a). The section is located in the southern part of the Netherlands in the Maastrichtian type area and can be correlated to nearby deposits in the Curfs quarry (figure 3.1). The Geulhemmerberg K-Pg boundary sediments (figure 3.2) were deposited in a shallow marine environment and the section is characterised by a succession of shallow water calcarenites in the top Maastrichtian deposits (unit IVf-6). The K-Pg boundary level is represented by an undulating hardground (the Berg and Terblijt Horizon), from which burrows extend into the underlying calcarenites. This conspicuous hardground is only found in this form in the Geulhemmerberg section; it changes laterally into the flat, burrowed surface that is recognised in the Curfs quarry. Above the hardground, storm and backwash deposits are found in the basal Danian (earliest Palaeogene), which was deposited in a marginal setting above wave base, with intercalated clay layers up to 15 cm thick (unit IVf-7) (Roep and Smit, 1996; Smit and Brinkhuis, 1996) (figure 3.2).

Like most other known K-Pg boundary sections that were deposited in a marginal setting, the Geulhemmerberg section contains a hiatus at the boundary. However, unlike in other sections, the time span contained in the hiatus in the Geulhemmerberg section is believed to be negligible (less than a hundred years) for various reasons, summarised by Smit and Brinkhuis (1996). Integrated sedimentological, geochemical, biostratigraphic and isotope studies point to an early Danian age of the strata immediately above the Berg and Terblijt Horizon, with the earliest Danian missing (Smit and Brinkhuis, 1996). However, C17¬ KP boundary biomarkers (a cyclopropyl fatty acid that is present in the K-Pg boundary clay in other sections) were found in burrows extending from the hardground (Yamamoto et al., 1996). Neither an impact layer nor a clear Ir anomaly have been found at the boundary, but the Ir/Th and Cr/Th ratios, which are elevated in the global ejecta layer, directly below the boundary in the uppermost Maastrichtian are slightly elevated, which could be the result of downward diffusion or transport from a once-present ejecta deposit (Smit and Rocchia, 1996).

If the interpretation of a very short to negligible hiatus is correct, the Geulhemmerberg K-Pg boundary section represents a very rare expanded earliest Palaeogene succession and could provide detailed information about the events immediately after the mass extinction. The

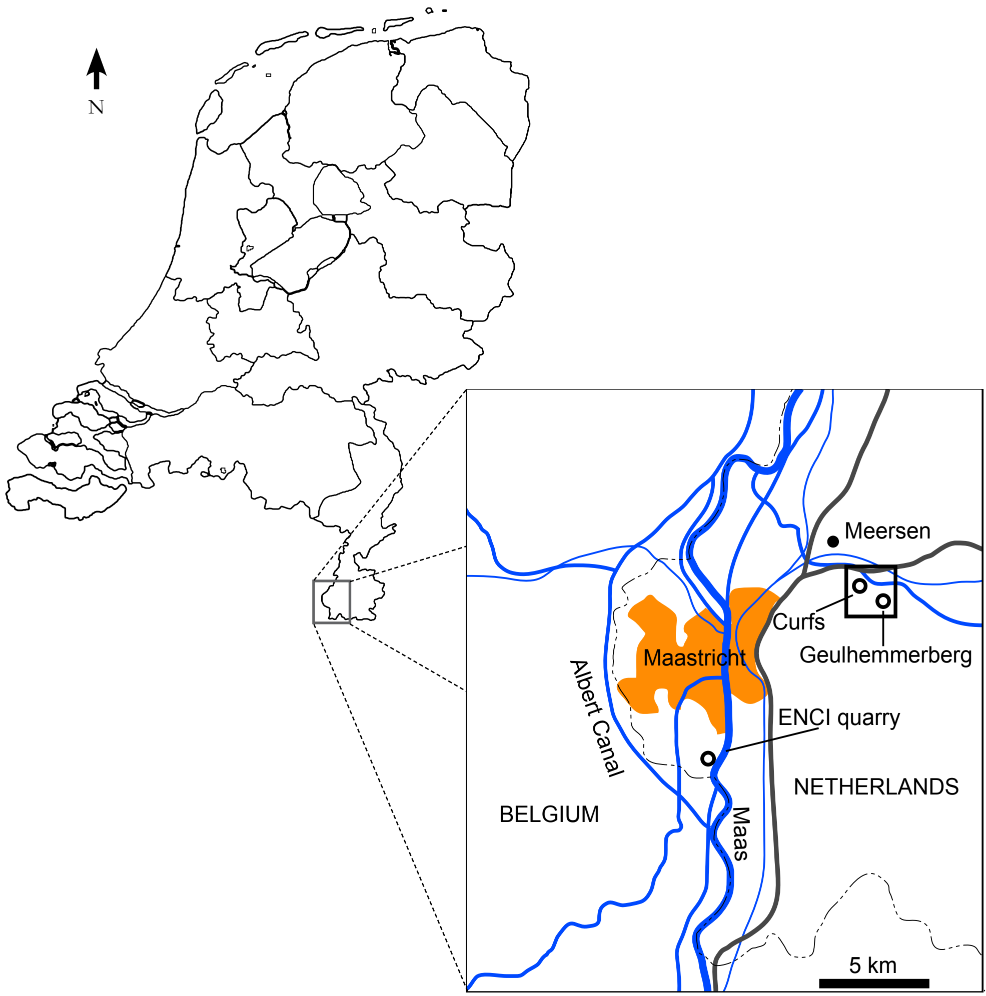

Figure 3.1 Location of the Geulhemmerberg caves (modified after figure 1 in Brinkhuis and Smit (1996b)).

presence of clay layers at and just above the boundary in such a marginal setting is unique. With the presence of potential boundary markers in the sediments and burrows directly below the boundary (elevated Ir concentration, C17 biomarkers), one might also expect shocked quartz grains to be found here. In order to test the hypothesis that the hiatus at the Geulhemmerberg K-Pg boundary is negligible, we checked for the presence of shocked quartz grains in burrows extending from the Berg en Terblijt Horizon (K-Pg boundary) using light microscopy and SEM-CL imaging. Shocked quartz grains from the K-Pg boundary in the Quagliotti (Italy) and Madrid Railroad (USA) sections were studied for comparison. Furthermore, this study serves as a practical test of the SEM-CL imaging technique as a quick and easy method to confirm the presence of PDFs and distinguish them from other, non-shock related, (sub-)planar microstructures in quartz, such as tectonic deformation lamellae and healed fractures.

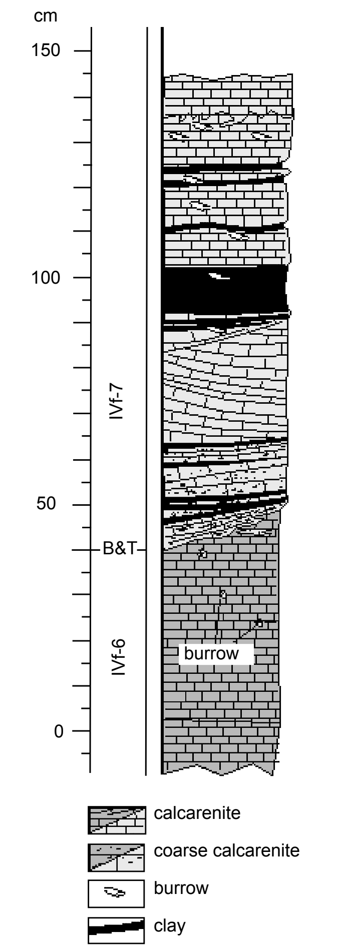

Figure 3.2 Simplified lithostratigraphic column of the Geulhemmerberg K-Pg boundary section (modified after figure 4 in Brinkhuis and Smit (1996b)). B&T indicates the K-Pg boundary level (Berg and Terblijt Horizon).

3.2 Samples and methods

Two sample sets were collected at the Geulhemmerberg K-Pg boundary: the first in 1996 (Brinkhuis and Smit, 1996a) and the second on May 12th, 2009 on the same site. Material was collected from burrows extending directly from the KP boundary clay layer.

Bulk samples were crushed if necessary, treated with HCl to remove all $CaCO_3$ and sieved to retain only the fraction with a grain size of 20-250 µm. Quartz grains were separated out of the bulk samples at the Mineral Separation Laboratory at the Free University of Amsterdam, using a Loc50 centrifuge with diiodomethane heavy liquid. For each sample three separations were done, with liquids of a density (d) of 2.66, 2.64 and 2.62 g/cm^3, resulting in four density fractions: 1) $d \leq 2.62$ g/cm^3, 2) 2.62 g/cm^3 $< d <$ 2.64 g/cm^3, 3) 2.64 g/cm^3 $\leq d <$ 2.66 g/cm^3, and 4) $d \geq$ 2.66 g/cm^3. Fractions 2 and 3 were embedded in epoxy (Epotek 301) in 0.5 inch diameter moulds and polished using a standard polishing procedure. Although quartz has a density of 2.65 g/cm^3, shocked quartz can have a lower density (Langenhorst and Deutsch, 1994). Therefore, both fractions 2 ($d = 2.63$ g/cm^3) and 3 ($d = 2.65$ g/cm^3) were studied.

17 samples, each containing hundreds of quartz grains, from the Geulhemmerberg were studied in a standard petrographic microscope. In total, 182 grains containing (sub-)planar microstructures were found, 130 of which were subsequently imaged using the SEM-CL techniques described in chapter 2. Two SEM systems with CL detector were used, both located

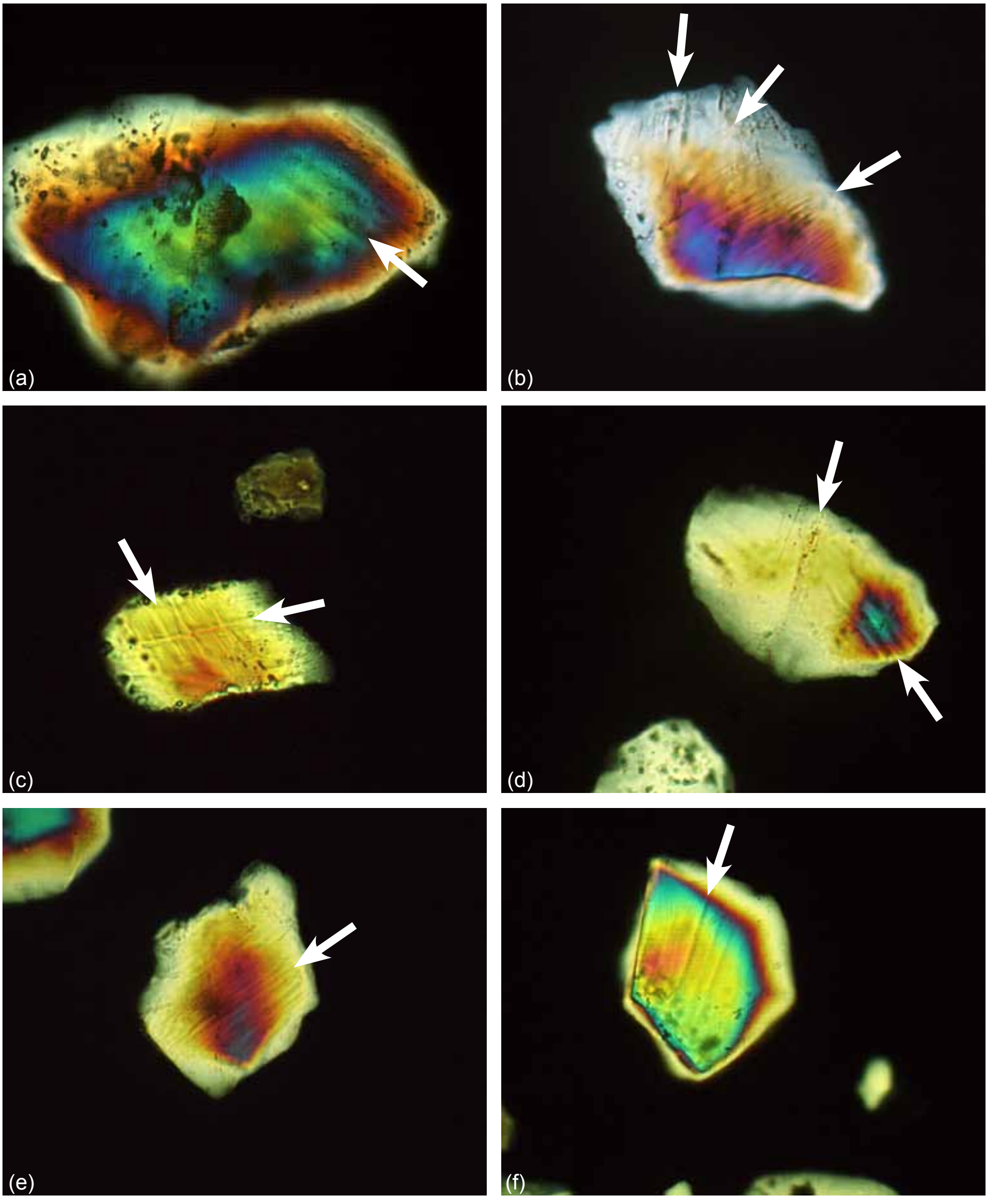

Figure 3.3 Light micrographs (crossed polars) of grains with (sub-)planar microstructures (white arrows) in quartz grains from burrows extending from the K-Pg boundary in the Geulhemmerberg section. Grains are between ~50 and ~100 μm in size. Images courtesy of Jurian Scholten.

at the Electron Microscopy Utrecht lab (EMU) at Utrecht University. The first is a Philips XL30S FEG-SEM with a KE Developments Centaurus CL detector attached, which has a wavelength detection range of 300-650 nm with a peak at 420 nm and is thus more sensitive to blue to violet light. The second system is an FEI Nova Nanolab 600 SEM with Gatan PanaCL detector, which is more or less panchromatic and has a detection range of 185-850 nm. Three colour filters (red, green and blue) can be fitted to the detector to record wavelength filtered images. The red filter primarily transmits light in the range 595-850 nm, the green filter in the range 495-575 nm, and the blue filter in the range 185-510 nm. Both unfiltered and filtered images were recorded; the filtered images were subsequently combined into composite RGB colour images. Because red, green and blue channels were optimised individually in order to obtain the maximum amount of information from the image, colour information in the images is non-quantitative. The instruments were operated at an acceleration voltage of 5-10 kV and with a beam current of 1.6-6.3 nA, at room temperature. Before SEM imaging, samples were carbon coated to prevent charging.

Of the 130 grains from the Geulhemmerberg section studied using SEM-CL, greyscale images were recorded of 68 and composite colour images of 62. Furthermore, for comparison, 32 shocked quartz grains from K-Pg boundary sites in Italy (Quagliotti quarry, ~10 km east of Ancona, Umbria (Montanari and Koeberl, 2000)) and the USA (Madrid Railroad outcrops, ~20 km west of Trinidad, Colorado (Izett, 1990)), where the ejecta layer is present, were imaged using composite colour SEM-CL, of which three grains were also studied in greyscale SEM-CL images.

3.3 Results

3.3.1 Geulhemmerberg quartz grains

(Sub-)planar microstructures in quartz were observed in 182 grains using light microscopy. Examples are shown in figure 3.3. Lamellae are indicated with white arrows. Most grains contain a single set, but rarely two or even three sets seem to be present (e.g. figures 3.3b, c and d). Only rarely lamellae were observed that were decorated with fluid inclusions (e.g. the NNE-SSW oriented lamellae in figure 3.3d).

Examples of SEM-CL images of quartz grains with lamellae are shown in figure 3.4. Two types of sub-planar microstructures can be distinguished in the SEM-CL images of these grains. Most striking are the non-luminescent, sharply defined, irregular semi-linear features in figures 3.4c and e (indicated by black arrows). Most of these non-luminescent features are not strictly straight and different orientations can occur in one grain. The most frequently observed type of lamellae is shown in figures 3.4a, b, d and e (indicated by white arrows). They are wide (often several µm in width), slightly curved features of varying thickness that can be either darker or lighter than the surrounding quartz in greyscale CL images (figures 3.4e and f), and range in colour from red to blue in composite colour CL images (figures 3.4 a, b and c). The two types of lamellae can occur together in one grain (see figure 3.4e).

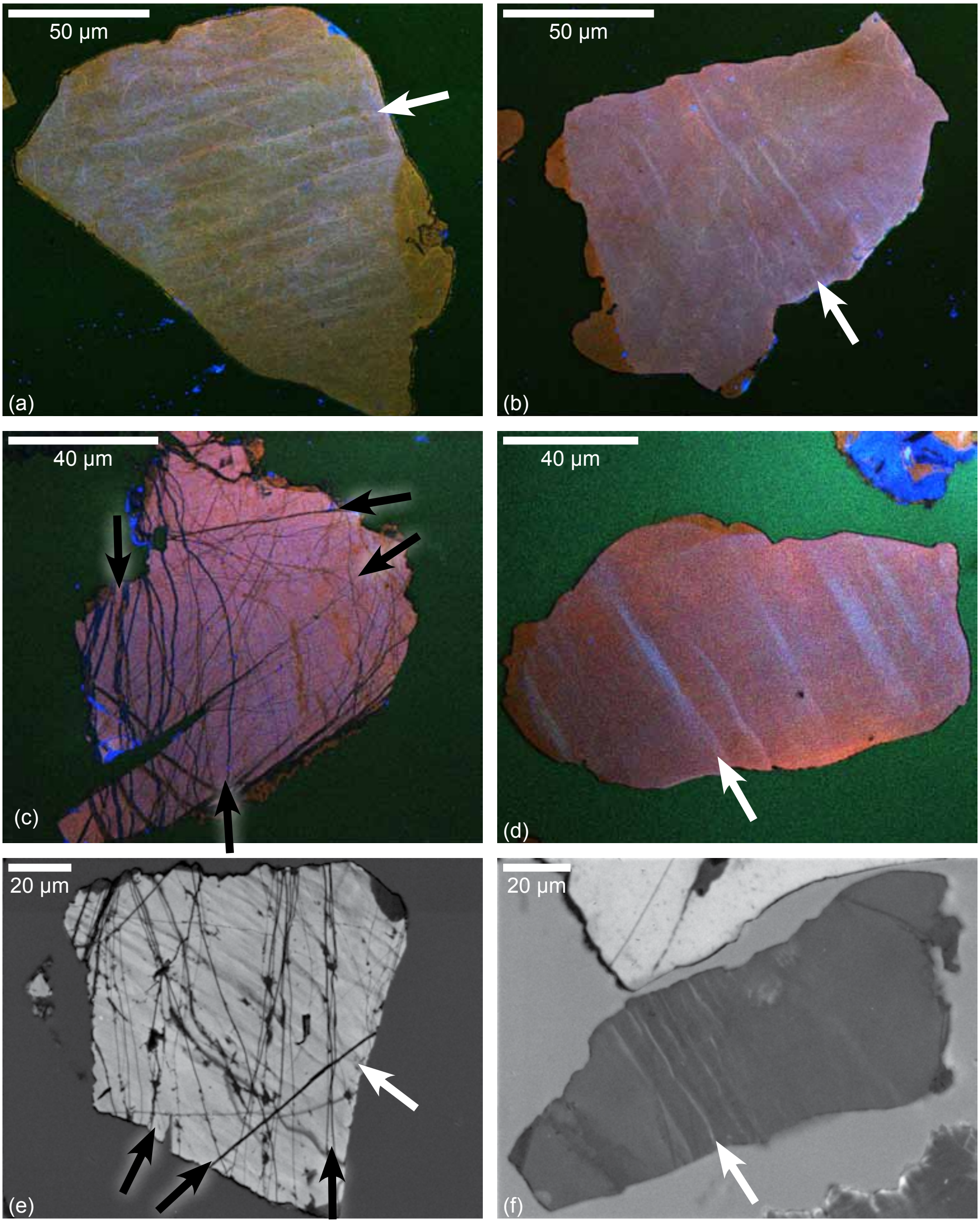

Figure 3.4 SEM-CL images of quartz grains from burrows extending from the Geulhemmerberg K-Pg boundary. **a-d** Composite colour SEM-CL images. **e-f** greyscale SEM-CL images. Two different types of microstructure are indicated by black and white arrows.

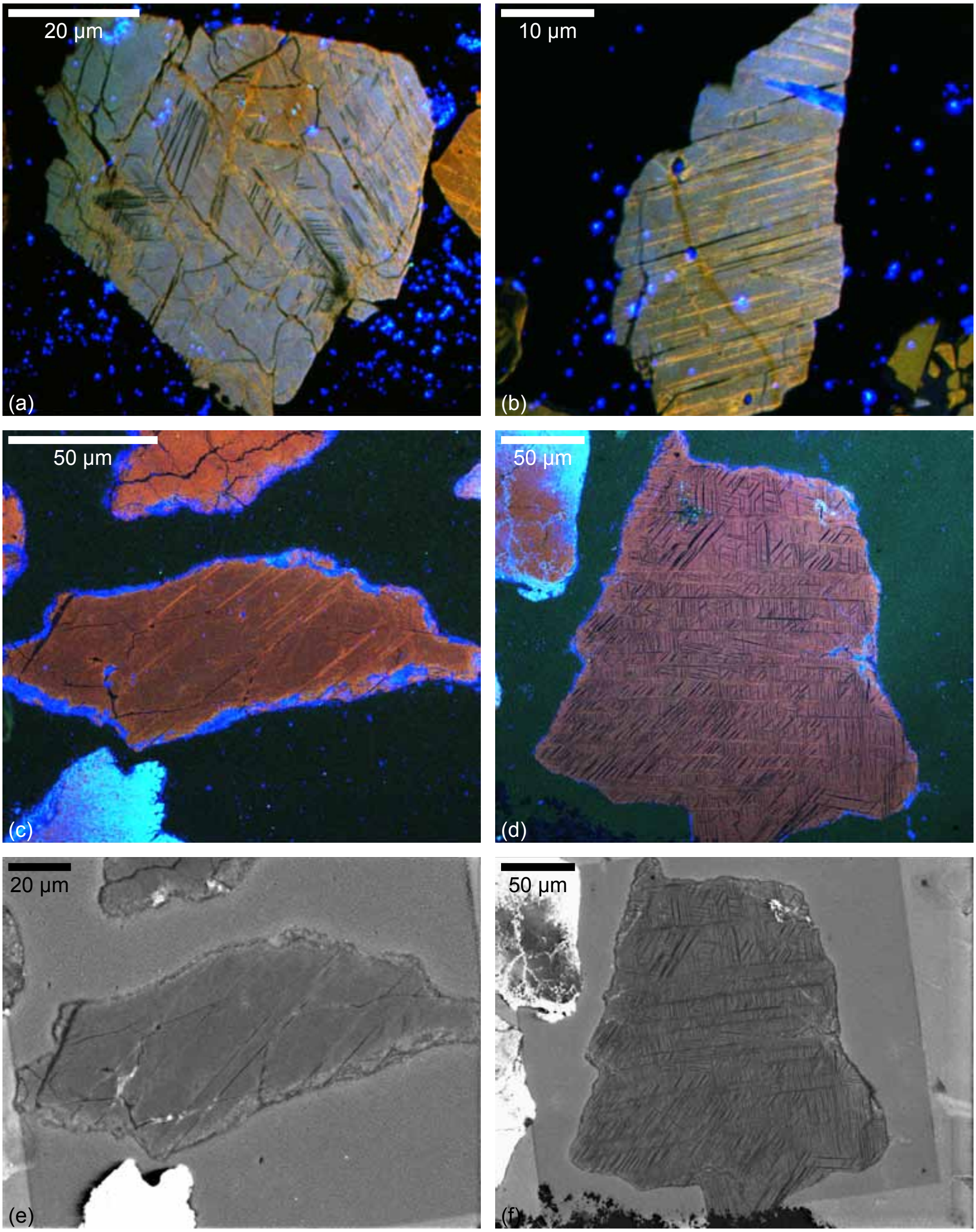

Figure 3.5 SEM-CL images of shocked quartz grains from the K-Pg boundary layers at the Quagliotti section (Italy) and Madrid Railroad section (USA). **a-b** Composite colour SEM-CL images of grains from the Quagliotti section in Italy. **c-d** Composite colour SEM-CL images of grains from the Madrid Railroad section, USA. **e-f** Greyscale SEM-CL images of the same two grains as in c and d. Bright blue spots (especially clear in 5a and 5b) are remains of polishing material on the sample surface.

3.3.2 Shocked quartz from other K-Pg boundaries

Figure 3.5 shows examples of SEM-CL images of shocked quartz grains with PDFs from the impact layer at K-Pg boundary sites in Italy (figures 3.5a and b) and the USA (figures 3.5c-f). The PDFs are thin (< 1 μm) and strictly planar, show no thickness variations and are red or non-luminescent. Some dark (parts of) PDFs are actually open or etched out instead of non-luminescent, but this distinction can only be made in secondary electron (SE) images showing surface topography. Especially in grains with multiple sets, the PDFs are closely spaced (1-2 μm). In greyscale CL images not all PDFs are clearly visible (figure 3.5e), but most are non-luminescent (figure 3.5f) or apparently non-luminescent. Apparent non-luminescence in greyscale CL images of features that are red in colour CL images can occur because the detector used for the greyscale images is relatively insensitive to red light. Usually multiple sets of PDFs are observed in one grain, but also single sets occur (e.g. figures 3.5c and d).

3.4 Discussion

In light microscopy, identification of the lamellar microstructures in quartz grains from the Geulhemmerberg K-Pg boundary section either as PDFs or tectonic deformation lamellae is not straightforward. The observed lamellae are not sharply defined and usually not penetrative through a whole grain, which would suggest they are tectonic deformation lamellae. On the other hand, two, or arguably even three, orientations were observed in single grains, which would suggest a shock origin of the lamellae. However, when comparing the SEM-CL images of the lamellar features in figure 3.4 to the PDFs in the grains in figure 3.5, it is clear that the microstructures in Geulhemmerberg quartz do not have the same characteristics as the PDFs in shocked quartz grains from Italy and the USA. The PDFs in figure 3.5 are straight and very thin, occur in multiple, closely spaced sets, and are either red or non-luminescent. These are clear characteristics of PDFs as described in chapter 2. The lamellae in the grains from the Geulhemmerberg, on the other hand, show mostly characteristics of tectonic deformation lamellae, also described in chapter 2: the lamellae are slightly curved, relatively wide features without clearly defined boundaries (figures 3.4a, b, d and f). CL colours range from red to blue and no more than two sets are observed in one grain. The non-luminescent irregular features (figures 3.4c and e) are healed fractures, which were described by Boggs et al. (2001), and are even more readily distinguished from PDFs in CL images by the highly irregular shape, thickness and spacing of the fractures.

Clearly, the sub-planar microstructures that are observed in the optical microscope in quartz grains from the Geulhemmerberg K-Pg section are not PDFs, but tectonic deformation lamellae and healed fractures, which are not shock-related. Therefore, no new evidence for the presence of the characteristic ejecta layer at the K-Pg boundary level could be found in the Geulhemmerberg section and no more evidence is available for the short duration of the hiatus at the boundary than was reported in earlier studies. It remains probable, but not certain, that the Geulhemmerberg section represents a rare expanded record of the latest Cretaceous and early Palaeogene. However, our results confirm the conclusion of chapter 2, that SEM-CL images form a reliable basis for the distinction of PDFs and non-shock related (sub-)planar microstructures in quartz. The technique can be applied to separated and embedded quartz grains, but also to standard polished thin sections or rock slabs. SEM-CL imaging can therefore be used to check the impact origin of both potential geological structures and stratigraphic levels.

3.5 Conclusions

Although using light microscopy (sub-)planar microstructures were observed in quartz grains from the Geulhemmerberg K-Pg boundary layer, no PDFs could be identified in SEM-CL images. Only tectonic deformation lamellae and healed fractures are present in the grains that were studied. We therefore conclude that no shocked quartz is present at or near the K-Pg boundary in the Geulhemmerberg section. The hypothesis that the hiatus at the boundary in this section is insignificant and that the section is therefore close to complete cannot be confirmed. The successful application of SEM-CL imaging techniques in this study confirms the potential of this method to prove or disprove the presence of PDFs in quartz.

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