Data archive

for

SINGLE QUARTZ GRAIN SEM-CL/OPTICAL MICROSCOPY PROVENANCE ANALYSIS

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INTEGRATED SINGLE QUARTZ GRAIN SEM-CL/ OPTICAL MICROSCOPE ANALYSIS

The integrated SEM-CL/optical microscopy technique takes nine steps, summarized here, assuming that sample collection is completed and uncovered thin sections are available:

- 1) Thin sections must not be covered by a glass cover slip and have to be inspected first with an optical microscope to check for polishing quality. Uneven surfaces will cause unwanted artifacts and disturbances while acquiring SEM-CL images. If necessary thin sections have to be repolished.
- 2) Randomly selected areas should be marked on the thin section with a felt tip pen to ensure that the same areas can be found again with the SEM and for the grain-by-grain comparison of SEM-CL and optical features later on. It helps to include an orientation marker on the slide.
- 3) Thin sections need to be carbon coated before SEM-CL analysis to allow discharge of electrons and avoid sample damage during electron bombardment. The carbon coating is very thin so the marked areas are easily visible and later optical microscopy is not compromised.
- 4) Depending on grain size (coarse to fine sand size) and sorting, it is necessary to take overview images (50x) to support orientation on the sample. For later grain-by-grain SEM-CL/optical microscopy analysis, it is best to take a series of close-up images at 100-150x magnification for coarse to medium grain sized samples or up to 200-300x magnification for fine grain sized samples. If a sample is poorly sorted a series of images at different magnifications may be necessary.
- 5) Mineral identification can be supported by EDS spot analyses in cases of uncertainty. This method allows distinction among quartz, feldspar, and other minerals in the sample on the basis of their chemical composition.
- 6) Information gained from SEM-CL analysis is documented with digital images, on which at least 100 quartz grains should be labeled with numbers for the integrated single-grain analysis. Labeling can be done with any photo editing program. It is helpful to keep a copy of the original digital SEM-CL images, because labels may cover some grains.
- 7) Optical microscopy provides information on extinction behavior (undulatory or non-undulatory), and poly- or microcrystallinity, following the approaches of Basu et al. (1975), Young (1976), and Tortosa et al. (1991). Furthermore, the shape and size of individual quartz grains should be noted.
- 8) Placing the optical microscope next to the computer with the labeled SEM-CL images allows direct comparison of individual quartz grains by each method. Thus information from optical microscopy can be directly integrated with information from SEM-CL analysis. Direct comparison reduces ambiguities in the identification of quartz types as some features such as mottled CL, zoning or undulose extinction can occur in several types of quartz grains.
- 9) All information from both techniques for each individual grain can be collected in a list using a spread sheet program, which enables calculating the frequency of occurrence of the different properties.

Steps six to eight can take over three hours per sample if 100 grains are analyzed. We found that 100 grains per sample are sufficient to distinguish major quartz types. It is necessary to increase this number to up to 300 grains per sample if a more detailed analysis is needed. This provenance technique works best for coarse- to medium-grained sand and sandstone, but is also easily applicable to fine sand sizes. However, the smaller the grains, the more difficult it becomes to distinguish between certain quartz types. For example volcanic quartz, vein quartz or disaggregated polycrystalline quartz may all show dark CL and non-undulose or weak undulose extinction. Therefore, we recommend the use of this technique for sand sized sediment only and mainly for coarse to medium grain sizes.

SEM-CL/petrographic analysis

7 Sept. 03 date:

sample number: 3171 stratigraphic unit:

Minesof

petrographer: Kari Bassett location: Moria

	SEM-CL characteristics/features						p	etrographic	characteri	stics/featur	es	
					healed	deforma-						
grain	homogen-		mottled or		micro-	tion	linear	single	polycryst-	polycryst-	micro-	embay-
number	eous light	dark	patchy	zoned	fractures	lamellae	features	crystal	alline <3	alline >3	crystalline	ments
1	X	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~
2	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~
3	X	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	Х
4	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~
5	~ ~ ~	~ ~ ~	Х	~ ~ ~	Х	Х	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~
6	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~
7 etc.	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	Х	~ ~ ~	~ ~ ~	~ ~ ~

petrographic characteristics/features			grain characteristics		comments			results						
	weak	strong												
straight	undulose	undulose	open		grain								quartz	grain
extinction	extinction	extinction	fractures	rounding	shape	grain size							type	number
~ ~ ~	Х	~ ~ ~	~ ~ ~	angular	spherical	med. sand	۲ ۲	~	~ ~	~	~ `	~	plutonic	1
Х	~ ~ ~	~ ~ ~	~ ~ ~	sub-ang.	spherical	med. sand	zoning	has s	oft rou	Inde	d cor	ners	volcanic	2
~ ~ ~	Х	~ ~ ~	Х	angl.	oblate	fine sand	~ ~	~	~ ~	~	~ -	~ ~	volcanic	3
~ ~ ~	~ ~ ~	Х	~ ~ ~	rounded	spherical	med. sand	~ ~	~	~ ~	~	~ -	~ ~	metamorphic	4
~ ~ ~	~ ~ ~	Х	~ ~ ~	sub-ang.	spherical	med. sand	~ ~	~	~ ~	~	~ -	~ ~	deformed plutonic	5
~ ~ ~	~ ~ ~	~ ~ ~	~ ~ ~	rounded	spherical	fine sand	~ ~	~	~ ~	~	~ ~	~ ~	chert	6
~ ~ ~	Х	~ ~ ~	~ ~ ~	sub-ang.	oblate	med. sand	zoning	has s	harp a	ingul	ar co	orners	vein	7 etc.

Cape Foulwind and Hohonu Batholith sample locations



Sample	Optical microscope	SEM-CL
HRS2	Large crystals with weak undulose extinction	Microcracks, some randomly oriented, Microfractures with preferred orientation.
HRS12	Large crystals with weak undulose extinction	Microcracks, some randomly oriented, Microfractures with preferred orientation.
HDP10	Large crystals with weak undulose extinction	Microcracks and microfractures, very little patchy/mottled CL
HDP9	Large crystals with undulose extinction, deformation lamellae	Microcracks, deformation lamellae, slightly mottled CL
HMT2	Large crystals with strong undulose extinction and subgrain boundaries	Some crystals with microcracks, some with patchy/mottled CL
HBM3	Mainly large, deformed crystals, some with deformation lamellae, strong undulose extinction, occasional mosaic quartz	Less microcracks/microfractures and deformation lamellae, some patchy/mottled CL, start of grain boundary formation
HSM1	Mainly recrystallized mosaic quartz, small grains with straight to weakly undulose extinction	Mainly dark CL, some newly formed grain boundaries of small mosaic quartz visible
TFQ1	Recrystallized mosaic quartz, small grains with mainly straight extinction	Relatively light gray CL for mosaic quartz. Grain boundaries visible, no microcracks or deformation lamellae

Table A1: Deutgam granodiorite, optical microscope and SEM-CL observations

Note: Samples are listed in order of the NE-SW transect shown in Fig. 1. SEM-CL analysis is based on grayscale CL. Results might vary with color SEM-CL.

Sample	Lithology	Optical microscope	SEM-CL
KM42	Granitic gneiss	Recrystallized mosaic quartz, small grains with mainly straight extinction	Black to dark gray CL response, no remnants of plutonic quartz visible
KM40	Granitic gneiss	>95 % recrystallized mosaic quartz, small grains with mainly straight extinction	Mainly black CL, some small areas with patchy or mottled CL
KM36	Granitic gneiss	Most quartz crystals recrystallized, few crystals with strong undulose extinction	Some remnants of plutonic quartz, but mainly dark CL
KM27	Granitic gneiss	Most quartz crystals recrystallized, few crystals with strong undulose extinction	Some remnants of plutonic quartz, but mainly dark CL
KM9	Granitic gneiss	Many quartz crystals recrystallized, few crystals with strong undulose extinction	Some remnants of plutonic quartz, but mainly dark CL
KM2	Granite	Many quartz crystals recrystallized, some larger crystals with strong undulose extinction	Large remnants of plutonic quartz, preferably oriented healed fractures
KM74	Granite	Most quartz crystals recrystallized, some larger crystals with strong undulose extinction	Some remnants of plutonic quartz, but mainly dark CL
KM5	Mylonitic granite	Almost all quartz completely recrystallized. Mosaic quartz, small grains with mainly straight extinction	Very small, unconnected remnants of plutonic quartz. >90% dark CL of quartz
KM8	Granitic gneiss	Majority of quartz recrystallized mosaic quartz, very few crystals with strong undulose extinction	Some, small remnants of plutonic quartz, but mainly dark CL
KM53	Granitic gneiss	Most quartz crystals recrystallized mosaic quartz, very few crystals with strong undulose extinction	Some, small remnants of plutonic quartz, but mainly dark CL

Table A2: Cape Foulwind and Siberia Bay optical microscope and SEM-CL observations

Note: Samples are listed in order of the NE-SW transect shown in Fig. 2. SEM-CL analysis is based on grayscale CL. Results might vary with color SEM-CL.

ADDITIONAL SEM-CL IMAGES

SEM-CL images in this data archive are only a very limited subset of the reference data base that we compiled during our study. These additional images are given here because they could not be all fit into the manuscript. Our complete reference data base is available in electronic from (as a Microsoft Access data base) upon request from the first author.

Plate 1 – plutonic quartz

- A) Plutonic quartz from Deutgam Granodiorite, Hohonu Batholith, South Island, New Zealand, showing microcracks and healed fractures. Note dark CL of biotite and brighter CL of feldspar. Very high luminescent spots in biotite and quartz are apatite and zircon inclusions.
- B) Same as A). Note so-called spiders (after Seyedolali et al. 1997; Kwon and Boggs 2002). Some microcracks show a broadly spaced pattern with a preferred orientation, which is an indication for tectonically induced deformation.
- C) Close-up image of B).
- D) SEM-CL image of plutonic quartz showing healed fractures in dark gray and microcracks as black lines.
- E) Further example SEM-CL image of plutonic quartz with typical healed fractures, microcracks and inclusions.
- F) More or less same location as E), seen with cross-polarized light in an optical microscope.

Plate 2 – volcanic quartz

- A) Volcanic quartz of Mount Somers rhyolite, South Island, New Zealand, showing open fractures, very weak zoning with lighter rim, somewhat patchy CL, and melt inclusion mainly in the center of the grain.
- B) Volcanic quartz from same location as A), with large open cooling-related fractures. The grain also shows an embayment, which contains melt material. Weak zoning visible.
- C) SEM-CL image of volcanic quartz from ignimbrite of the Tarawera Volcanic Complex, North Island, New Zealand. Similar to the previous images this grain contains large open fractures that are filled with epoxy and appear white, and also inclusions and embayments with melt material.
- D) Volcanic grain of same location as C), showing melt inclusion, but otherwise fairly homogeneous CL.
- E) Volcanic quartz of Mount Somers rhyolite, collected at Rakaia Gorge, South Island, New Zealand, with relatively dark CL, partly an optical effect because of the bright surrounding matrix. The grain shown very weak zoning with a fairly homogeneous core, surrounded by a zone of patchy CL and a bright CL rim.
- F) Another example of volcanic quartz with relatively dark CL from Mount Somers ignimbrites, South Island, New Zealand. The grain shows inhomogeneous CL and melt inclusions.



Plate 1 - SEM-CL images of plutonic quartz - Bernet and Bassett 2004



Plate 2 - SEM-CL images of volcanic quartz - Bernet and Bassett 2004

Plate 3 – deformed and recrystallized quartz

- A) SEM-CL image of partially recrystallized plutonic quartz in granitic gneiss of Cape Foulwind, Western Province, South Island, New Zealand. Non-recrystallized parts still show microcracks and healed cracks, while recrystallized domains appear black in SEM-CL images.
- B) Same phenomenon as in A), observed in formerly plutonic quartz of granitic gneiss from Siberia Bay, Western Province, South Island, New Zealand.
- C) Overview image of partially recrysatllized quartz from same location as B).
- D) Optical microscope image of recrystallized quartz (mosaics) of same sample as C). Note areas of recrystallized quartz (circle).
- E) Example of plutonic quartz from the Deutgam Granodiorite showing microcracks as well as narrowly spaced wavy deformation lamellae.
- F) SEM-CL image of quartz with relatively dark and patchy CL. This grain shows relatively strong undulose extinction under a polarizing microscope.

Plate 4 – vein quartz and microcrystalline quartz

- A) SEM-CL image of narrowly spaced oscillatory zoned quartz in vein quartz from Te Puru Creek, Coromandel Peninsula, North Island, New Zealand.
- B) Secondary vein in larger vein quartz, showing some zoning of individual crystals.
- C) Recrystallized vein quartz from Otago schist, South Island New Zealand, appearing mainly black in SEM-CL image, and somewhat patchy.
- D) SEM-CL image of microcrystalline chalcedony from Te Puru Creek, Coromandel Peninsula, North Island, New Zealand.
- E) SEM-CL image of vein quartz from the Wepawaug Schist, western Connecticut, USA, showing relatively dark, patchy CL.
- F) Further example of an SEM-CL image of vein quartz from the Wepawaug Schist, western Connecticut, USA. This sample display somewhat patchy CL but also the very rare occurrence of microcracks in vein quartz.



Plate 3 - SEM-CL images of deformed and recrystallized quartz - Bernet and Bassett 2004



Plate 4 - SEM-CL images of vein and microcrystalline quartz - Bernet and Bassett 2004

Sample locations of quartz bearing lithologies for SEM-CL reference data base



Samples for SEM-CL analysis of quartz

List A

No.	Field #	Lithology	Location	Comment
1	HRS2	granodiorite	Deutgam Granite, NZ	
2	HRS12	granodiorite	Deutgam Granite, NZ	
3	HDP9	granodiorite	Deutgam Granite, NZ	
4	HDP10	granodiorite	Deutgam Granite, NZ	
5	HSM1	granodiorite	Deutgam Granite, NZ	
6	HBM3	granodiorite	Deutgam Granite, NZ	
7	HMT2	leucogranite	Deutgam Granite, NZ	
8	TFQ1	granodiorite	Deutgam Granite, NZ	
9	KM9	gneiss granite	Cape Foulwind Granite, NZ	
10	KM27	gneiss granite	Cape Foulwind Granite, NZ	
11	KM36	gneiss granite	Cape Foulwind Granite, NZ	
12	KM40	gneiss granite	Cape Foulwind Granite, NZ	
13	KM42	gneiss granite	Cape Foulwind Granite, NZ	
14	KM2	granite	Siberia Bay Granite, NZ	
15	KM5	mylonitised granite	Siberia Bay Granite, NZ	
16	KM8	gneiss granite	Siberia Bay Granite, NZ	
17	KM53	gneiss granite	Siberia Bay Granite, NZ	
18	KM74	granite	Siberia Bay Granite, NZ	
19	95 AZ 507	ignimbrite	Mustang Mts., Camelo Hills volc.	
20	95 AZ 509	ignimbrite	Arizona	
21	95 AZ 524 SR	granite	Squaw Gulch Granite, US	
22	95 AZ 526	conglomerate	Glance congl., Camelo Hills, US	
23	95 AZ 529a CH	ignimbrite	Camelo Hills, US	
24 95 AZ 535		ignimbrite	Arizona	
25 96 AZ 802		tuff	Arizona	
26 96 AZ 659a		tuff	Arizona	
27 95 AZ 510		tuff/ignimbrite (?)	Arizona	
28	95 AZ 520 d1	tuff/ignimbrite (?)	Arizona	
29	SCL2	volcanic	Antarctica	
30	SCL5	plutonic	Antarctica	
31	CAC 1/1	gneissic	Antarctica	
32	CAC 1/8	granitic	Antarctica	
33	CAC 1/10	gneissic	Antarctica	not analyzed
34	CAC 2/1	granitic	Antarctica	
35	CAC 2/3	gneissic	Antarctica	
36	CAC 4/4	volcanic	Antarctica	
37	CAC 5/2	volcanic	Antarctica	
38	MS221	rhyolite	Mount Somers, NZ	
39	MS218	rhyolite	Mount Somers, NZ	
40	MS225	rhyolite	Mount Somers, NZ	
41	MS223	rhyolite	Mount Somers, NZ	
42	MH3	ignimbrite	Mount Somers, NZ	
43	MH7	ignimbrite	Mount Somers, NZ	
44	MS99	pitchstone	Mount Somers, NZ	
45	MS216a	pitchstone	Mount Somers, NZ	
46	MS216b	pitchstone	Mount Somers, NZ	not analyzed
47	JAW 113E	vein quartz	Wepawaug Schist, CT, USA	
48	JAW -1	vein quartz	Wepawaug Schist, CT, USA	
49 JAW-30Da2		vein quartz	Wepawaug Schist, CT, USA	
50	SC	quartz arenite	Upstate NY, USA	
51	SP90-1	quartz arenite	Upstate NY, USA	
52 A1		quartz arenite	Upstate NY, USA	

Samples for SEM-CL analysis of quartz

List B

No.	Field #	Lithology	Location	Comment
53	A4a	rhyolite	Tarawera volcanic complex, NZ	
54	H102	granodiorite	Tarawera volcanic complex, NZ	
55	IAN3	rhyolite	Tarawera volcanic complex, NZ	
56	A1a	rhyolite	Tarawera volcanic complex, NZ	
57	LH1	schist/vein quartz	Otago	
58	LH11	schist/vein quartz	Otago	
59	LH6b	schist/vein quartz	Otago	
60	LP2	schist/vein quartz	Otago	
61	HP4	schist/vein quartz	Otago	
62	02MB60	mylonite	near White Horse Creek (?), NZ	
63	02MB61	granite	Meybille Bay, NZ	
64	02MB62	rhyolite	Rakaia River Gorge, NZ	
65	02MB63	ignimbrite	near Lichfield	
66	02MB64	pumice	Whakamaru	no quartz
67	02MB65	pumice	Turangi	no quartz
68	02MB68	ignimbrite	Kuratau	
69	02MB69	sand	Shore of Lake Taupo	
70	02MB71	dacite	Mt. Tauhara	
71	02MB72	dacite	Mt. Tauhara	
72	02MB73	dacite	Mt. Tauhara	
73	02MB74	rhyolite	Haroharo volcanic complex	
74	02MB75	rhyolite	Haroharo volcanic complex	
75	02MB76	ignimbrite	Haroharo volcanic complex	
76	02MB77	dacite	Haroharo volcanic complex	
77	02MB78	dacite	Haroharo volcanic complex	
78	02MB79	sand	Shore of Lake Okataina	
79	02MB80	sand	Beach of Bay of plenty	
80	02MB81	dacite	Waihi	
81	02MB83	dacite	Waihi area	
82	02MB84	sand	Hotwater beach	
83	02MB85	veins in greywacke	Kuaoturu, Coromandel	calcite
84	02MB86	veins in greywacke	Kuaoturu, Coromandel	calcite
85	02MB91	vein quartz	Te Puru Creel, Coromandel	
86	02MB92	vein quartz	Te Puru Creel, Coromandel	
87	02MB94	chalcedony	Kauaeranga river valley	
88	02MB95	jasper	Kauaeranga river valley	
89	02MB96	sand	Tapotupotu Bay, Cape Reinga, NZ	
90	03MB100	greensand	Island Hills	
91	03MB101	vein quartz	Franz Josef Glacier	
92	03MB102	vein quartz	Franz Josef Glacier	
93	03MB104	vein quartz	Haast Pass	
94	03MB105	vein quartz	Haast Pass	
95	03MB106	sandstone clast	Rakaia river gorge	
96	03MB110	quartz sand	Mt Somers	
97	03MB111	quartz sand	Mt Somers	
98	03MB113	quartz sand	Mt Somers	
99	03MB114	quartz sand	Mt Somers	
100	03MB116	quartz sand	Mt Somers	
101	03MB118	quartz sand	Mt Somers	
102	03MB121	greenland group	12 mile bluff	
103	03MB122	vein quartz	12 mile bluff	
104	03MB126	quartz arenite	Brunner coal measures	
105				