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The preservation of quartz grain surface textures following vehicle fire and their use in forensic enquiry

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Abstract

During a terrorist trial, dispute arose as to whether the temperature produced in a car fire was sufficient to destroy quartz grain surface textures. A series of seven sequential experiments showed that the temperature for quartz surface texture modification/destruction and the production of vugs, vesicles and glassy precipitation ('snowdrifting') occurred at 1200°C under normal atmospheric conditions. By adding a number of man-made and natural substances, it was found that only the presence of salts depressed this modification temperature (to 900°C). Experiments to determine the temperature of fire in a car indicated that the maximum temperature produced under natural conditions (810°C) was insufficient to affect the quartz grain surface textures. These results confirm the use of surface texture analysis of quartz grains recovered from the remains of cars subjected to fire and their use as a forensic indicator.

Keywords: Forensic science, quartz, vehicle fire temperatures, scanning electron microscopy.

1. Background

During a trial involving a terrorist attack, the prosecution presented, as part of their evidence, the results from quartz grain surface texture analysis of various soils from the attack scene and from associated comparator sites. Some of these quartz grains had been recovered from a vehicle which had been completely destroyed by fire, and consequently no other physical evidence (such as DNA, fibres, pollen etc.) could be recovered. The defence argued that this geological evidence should be disregarded, claiming that the temperatures experienced during the fire would have caused the surviving quartz grains to become so heavily fractured and altered that their surface characteristics would no longer be recognisable. This paper therefore, sets out to determine under what conditions quartz grain surface textures can be preserved after exposure to the temperatures associated with vehicular fire.

2. Introduction

Quartz is a highly resistant and therefore, a ubiquitous mineral found in most soil/sediment samples. It has been shown to demonstrate a range of clearly identifiable and environmentally specific surface textures [1-5] containing texture assemblages which form specific grain types [6-9]. Recent studies show the relevance of this technique as a 'fingerprinting mechanism' useful in the forensic arena [10].

Soil/sediment samples have been analysed and compared for forensic purposes by a number of methods including colour [11-12], organic content [13], biological characteristics [14-16], particle size analysis [17-20] and mineralogy [21]. However, the action of fire will have a profound influence on the viability of all these measurable characteristics of soil as significant modifications to these characteristics occur. Colour and particle size distributions of a soil have been shown to be modified at 400^{o}C [22, 23], as have mineralogy [24] and soil/sediment chemistry [25]. Heating initially affects the biological activity of a soil/sediment, while intermediate temperatures from sustained heating can implement appreciable chemical changes. When temperatures reach sufficient levels, permanent physical alterations occur (as outlined in figure 1 [26]).

Fires and their associated high temperatures can therefore, have a multitude of impacts on the chemical, physical and biological characteristics of a soil/sediment sample rendering many of the traditional analytical techniques employed in forensic analysis impracticable under these conditions. It has however, been observed that after severe burning quartz, mica and feldspar grains survived [23]. It is therefore important to know whether the quartz grain surface textures are modified or altered after exposure to high temperatures rendering this technique equally unusable in the situations such as that found in vehicle fires.

This paper presents a series of experiments which have been undertaken to assess the impact of temperature on quartz grain surface textures, and to offer conclusions in the light of this work as to the practicability of this analytical technique for forensic soil/sediment samples that have been subjected to high temperature environments. The experiments involve ascertaining the critical temperature for quartz grain surface modification, whether this critical temperature varies according to different types of quartz and whether it varies in the presence of other materials normally associated with vehicles and soils (catalysts or inhibitors).

3. Experimental Work

3.1 Experiment 1

The aim of this experiment was to observe the critical temperature of quartz surface texture modification under normal atmospheric conditions using heat generated from a muffle furnace. Four samples were used which comprised of two types of soil and two types of sand (Soils A and B taken from Oxfordshire and Coventry respectively, aquarium sand (sand C), and Chilean coastal dune sand (sand D)). These different types were chosen because of their varied origin and hence their varied characteristics. A control sample from each type of soil and sand was cleaned by boiling 5g of each sample in a test tube with sodium-hexametaphosphate, followed by washing with distilled water (for cleansing and the removal of soluble salts) and repeating the process with 10% hydrocholoric acid. The sample was then dried in an oven at 40^{o}C and gently disaggregated. Medium sand-sized quartz grains were then selected and one hundred of these ‘control’ grains from each sample were analysed using the scanning electron microscope (SEM). Characteristic surface texture features were noted and grain types established [10].

One gram of each sample was then taken and heated in a high-heat tolerant alumino-silica crucible at 900^{o}C for 30 minutes in a muffle furnace. The soil samples were washed to remove any charred debris, and 30 quartz grains extracted and mounted on SEM aluminium stubs for surface texture analysis. This procedure was also repeated on samples which were heated to 1200^{o}C.

The surface textures of the quartz grains in samples A, B and C which were heated to 900^{o}C, exhibited no physical change before or after heating (0% of the grains were modified). Chi-squared results confirmed that soils A and B and sand C heated at 900^{o}C were not significantly different (at the 95% significance level) to the equivalent unheated samples. However, sand D was found by Chi-squared analysis to be significantly different (indeed 90% of grains were modified). In order to assess why sand D may have behaved significantly differently to samples A, B, and C at 900^{o}C, Dionex analysis was undertaken on the soluble constituents of soils A and B, and sand D. This test revealed a high salt content in sand D (on average 527.34ppm chloride, 56.42ppm sulphate where n=3) in comparison to the other samples. The possible impact of the presence of salt is explored further in Experiments 5 and 6 of this paper.

The surface textures of the grains that were subjected to a temperature of 1200^{o}C exhibited notable changes, with the appearance of vugs (figure 2), cracks [27] (figure 3) and glassy precipitation (‘snowdrift’) (figure 4). It was found that 83% of the quartz grains in soil A were modified, 60% in soil B, 93% in sand C 100% in sand D. These identifiable changes at 1200^{o}C made it impossible to identify the surface texture of the quartz grains and, in effect, rendered it impossible to determine that these altered grains contained the same characteristics as those of the control samples. Thus, at 1200^{o}C all samples revealed quartz grain surface modification whilst at 900^{o}C the quartz surfaces from samples A, B and C remained unmodified. Quartz from sample D did however exhibit modification at 900^{o}C which may be due to an elevated salt content of the sample.

3.2 Experiment 2

This experiment was formulated to test the conclusions derived from experiment 1 and to include the consideration of the effect of the introduction of denim, cotton and lycra materials as having a possible catalytic or inhibiting effect on the quartz modification critical temperature. The same procedure as used in experiment 1 was repeated on the same four sand and soil samples which were this time wrapped in material swatches.

The results of these experiments demonstrated that the addition of denim, cotton or lycra to the 30 quartz grains studied appeared to have no effect on the occurrence of modification of the quartz grain surfaces at 900^{o}C and 1200^{o}C. The quartz grains which were heated to 900^{o}C still resembled the quartz in the corresponding control sample (figures 5, 6 and 7) (none of the grains from soil A and B were modified, and only 0.01% were modified in sand C). At 1200^{o}C, the quartz surface textures was still shown to be statistically different (Chi-squared analysis) to the corresponding control sample (with 100% of soils A and B and sand C quartz grain surfaces modified). Sand D still demonstrated modifications at a lower temperature threshold, with 88% of quartz grain surfaces modified at 900^{o}C and 100% modified at 1200^{o}C. In conclusion therefore, the results of experiment 1 and experiment 2 can be judged as having a similar outcome.

3.3 Experiment 3

Further experiments were undertaken with five additional sand samples which all had a high quartz content (soft white sand from Lochaline, West Scotland; sand from Cheltenham; sand from Storsjoen Lake, East Norway; beach sand from Douglas, Isle of Man; beach sand from Keem Bay, Archill Island, Co Mayo). These samples were used to test the possible catalytic effect of engine oil, car body paint, rubber, footwell carpet and plastic respectively. Firstly, the samples were subjected to temperatures (in a muffle furnace) at 200^{o}C increments between 200^{o}C and 1200^{o}C to establish whether the quartz surfaces in these samples behaved similarly to previously tested samples (i.e. no change at 900^{o}C, but with modifications apparent at 1200^{o}C). Quartz surface texture analysis was carried out on the untreated 'control' grains from each sample, and the results compared with SEM analysis of the grains subjected to 200^{o}C, 400^{o}C, 600^{o}C, 800^{o}C, 1000^{o}C and 1200^{o}C temperatures with a catalyst (as mentioned above).

These experiments on sand from five different sources to that used in experiments 1 and 2 confirmed that a temperature threshold of 1200^{o}C does indeed exist at normal atmospheric pressures for the modification of quartz surface textures. In addition, the results of these experiments agree with the results obtained in experiment 2 that the presence of other materials, in this case engine oil, car body paint, rubber, footwell carpet and plastic, appears to have no effect on the modification of the quartz grain surface textures.

3.4 Experiment 4

Having established the premise that in general quartz surfaces are modified at 1200^{o}C (with the exception of the marine sand in experiments 1 and 2 – for further discussion see 3.5), and that various clothing items, paints, oils and plastics have no effect upon this critical temperature, this series of experiments examined the effect of direct vehicular fire upon the sand grains. Three experimental vehicle fires were performed at Moreton in Marsh Fire Service College (in conjunction with a forensic arson investigation course) to assess their impact on quartz grain surface textures. These three scenarios involved:

1. Sierra XR4i: an engine fire started using wood and fire lighters.
2. Ford Escort: a rear seat cabin fire started using a cigarette lighter and a crisp packet.
3. Renault 5: a simulated electrical fire started in the driver's side dashboard using a fire lighter.

Sand samples (from the same sources as those used in experiment 3) were positioned within the cars in the left and right front and back footwells and the left and right front wheel arches. Each location had samples which had been prepared in both ‘open’ and ‘closed’ combustion boats. The closed combustion boats did not expose the quartz to naked flame, but ensured that some samples survived the blast of the fire hoses. Thermocouples were attached to the driver side footwell and left and right wheel arch of each vehicle and each thermocouple was linked to a computer in the mobile control centre which was programmed to record the temperature every three seconds for the duration of the fire. Each fire was allowed to reach a peak fire intensity and a high level of cabin destruction.

It was found that none of the simulated vehicle fires achieved the critical temperature of 1200^{o}C (see figures 8 and 9). It is significant that the temperatures reached in these vehicular fires did not exceed 810^{o}C when considering the use of quartz grain surface texture analysis of quartz recovered from such situations. All of the quartz grains recovered demonstrated no sign of temperature-induced modification.

3.5 Experiment 5

This experiment was designed to investigate whether quartz formed by different mechanisms [28], which were submitted to the same procedures and temperatures as found in the previous experiments, exhibited similar alteration patterns. Four different types of quartz (crushed pegmatitic quartz, metamorphic schist, washed aeolian-derived sand and unwashed marine sand (the latter of similar origin to sand D in experiment 1 and therefore containing a high salt content)) were exposed to temperatures in the muffle furnace of 900^{o}C and 1200^{o}C. After SEM assessment of these initial results, further experiments were carried out (see figure 10) to assess if the length of time that the quartz grains were subjected to high temperatures, the particle size of the quartz, or the presence of salt (in the marine quartz sample) affected the modification of the quartz grain surface textures.

At 900^{o}C the pegmatitic quartz, the metamorphic schist and the aeolian derived quartz showed very little (but some) evidence of surface texture modification (0%, 4%, 0% of grains modified respectively) when compared to the corresponding untreated quartz grains. The marine quartz however displayed significant modification with 70% of the grains exhibiting modification such as cracks, glassy precipitation (snowdrift), and small vugs.

At 1200^{o}C, the metamorphic schist, washed aeolian and unwashed marine quartz all exhibited 100% modification, exhibiting features such as cracks, small vugs, glassy precipitation (snowdrift), single vesicles and large vugs (figures 2 and 7). However, only 4% of the pegmatitic quartz grains were so affected.

Due to the unexpected results encountered for the experimental run at 900^{o}C for the unwashed marine quartz and at 1200^{o}C for the pegmatitic quartz, further experiments were undertaken as outlined in figure 10.

The particle size of the pegmatitic quartz, and length of time of heating are not significant factors that affect the propensity of the quartz surface texture to modification at 1200^{o}C. However, the presence of salt in the form of sodium and magnesium chloride does appear to have an affect on quartz grain surface texture modification, as significant modification occurs to the quartz at 900^{o}C when this salt is present which does not occur when it is not present (see also results for sand D in experiments 1 and 2).

3.6 Experiment 6

Given the observation from experiment 5 that salts do indeed seem to have the same effect of reducing the critical temperature for quartz surface texture modification (as found in experiment 1), this experiment tested whether various salt solutions of differing complexity would also affect the critical temperature for quartz grain surface texture modification.

Fresh quartz grains were produced by crushing a large intact quartz crystal (as in experiment 5). The ‘control’ grains were heated at 800^{o}C, 900^{o}C, 1000^{o}C and 1100^{o}C, as were grains treated with sea salt solution precipitated onto the surface. This was then repeated with different types of salt (table salt pH 10.14, sea salt pH 9.30, Chilean salt (from the Atacama desert) pH 6.02 and Namibian salt (from the Namib desert) pH 6.51), at temperatures of 800^{o}C, 900^{o}C and 1000^{o}C.

These results confirm the findings of experiment 5 that the presence of salt depresses the temperature at which heat-induced modifications to surface textures are generated from 1200°C to 900°C. The surface textures of fresh, clean quartz crystals were not modified by temperatures up to 1100°C, however, in the presence of salt, heat-induced surface textures of the quartz occurred at 900°C (see figures 11 and 12).

The quartz grains treated with table salt demonstrated the most widespread heat-induced surface texture features at 900°C and 1000°C with all the grains exhibiting glassy precipitation (snowdrifting), and all but one grain exhibiting single vesicles. At 1000°C the single vesicles appear to have burst to produce the extensive vesicular structure effect on 27% of the grains. The quartz treated with sea salt showed a similar pattern, but no extensive vesicular features were seen. The quartz grains coated with Chilean and Namibian salt produced slightly different results. While there was significant glassy precipitation, no single vesicles occurred on the quartz grains, yet cracks [27] occurred on the quartz treated with Chilean salt and extensive vesicular structure features occurred on the quartz treated with Namibian salt.

3.7 Experiment 7

The last experiment in this series was constructed to determine whether soil or sediment pH played any part in affecting the critical temperature of quartz grain surface texture modification. Different types of soil were used (peat pH 3.24, topsoil pH 6.51 and limed topsoil pH 7.34). The soil samples were washed to test the pH and conductivity, and quartz grains (similar to those used in experiment 6) were then covered by each solution and placed in an oven for 30 minutes at 100°C to evaporate the excess water and precipitate minerals in the soil solutions onto the quartz grains. The quartz-soil mixtures were then subjected to temperatures of 800°C, 900°C and 1000°C.

It is pertinent to note that during these experiments, upon the removal of samples from the furnace, the 'catalysts' had all undergone physical change. All the peat burnt off leaving very clean looking quartz grains. The limed soil underwent only minor change, with the soil colour appearing darker after heating. The topsoil became pink, leaving a pink residue on the quartz grains after heating. Significantly, no heat-induced changes to the quartz grain surfaces were observed at 800^{o}C, 900^{o}C or 1000^{o}C. Therefore, pH does not appear to have any effect on the modification of quartz grains due to heat.

4. Conclusions

From these experiments, a number of conclusions can be drawn:

1. Under normal conditions, a temperature threshold of 1200^{o}C exists for the modification of quartz surface textures.
2. Quartz of different modes of formation demonstrate similar behaviour of a 1200^{o}C threshold for surface texture modification.
3. The heat-induced quartz grain surface textures that are observable at temperatures of 1200^{o}C and above include glassy precipitation (snowdrifting), single vesicles and extensive vesicular structures (figures 2, 3, 4 and 7). These features are similar to those identified in natural pyroclastic rocks [27].
4. Potential ‘catalysts’ such as clothing (cotton, denim and lycra), and substances present in motor vehicles (engine oil, car body paint, rubber, footwell carpet, and plastic) do not appear to have any effect on this threshold.
5. Vehicular fires (of small vehicles in this experimental study) do not achieve the temperature threshold of 1200^{o}C and so quartz grains recovered from such fires do not appear to have any heat induced modifications.
6. The pH of a soil/sediment does not appear to have any effect on the threshold temperature of quartz surface texture modification.
7. The presence of salt does appear to act to depress the temperature at which heat-induced quartz surface textures are generated from 1200^{o}C to 900^{o}C.
8. Salt does not have a uniform effect on quartz. Glassy precipitation (snowdrift) features are common to all kinds of salt tested, but single vesicles, extensive vesicular structures and cracks only occur in the presence of specific types of salt compositions.

It is well documented that when soil/sediment samples are subjected to high temperatures, significant modifications will occur to the colour, mineralogy, particle size, chemistry, organic content and biological characteristics of the sample. Quartz grains are one of the only components of a soil left after exposure to severe burning, alongside mica and feldspar grains [23], and therefore these findings have significant implications for the use of quartz grain surface texture analysis in forensic investigations. Temperature measurements taken from deliberately torched cars have shown that the maximum temperature reached in the car was 810^{o}C in the footwell, whilst wheel arch temperatures ranged from 285-735^{o}C. Therefore, the surface textures of quartz grains recovered from burning vehicles are highly likely to be unmodified by the heat (even when salt is present) and so can be considered suitable for forensic comparison with quartz grains that have not been heated. However, caution should be exercised when analysing quartz from marine or saline environments, as the presence of salt may affect the surface textures if the temperature reached the critical threshold of 900^{o}C. It is noted that these heat-induced textures are highly recognisable and characteristic, and therefore, only if they are present should the analysis and interpretation of quartz grain surface textures be adjusted accordingly.

The usual caveat of any forensic analytical technique applies in this case. As part of a suite of independent analytical techniques, this technique has the potential to be highly informative, reliable and accurate in making comparisons between forensic samples, and identifying where exclusion of a sample is possible.

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List of Figures

Dominant type of change	Temperature (^oC)	Change to the soil/sediment
Physical	>1200	Loss of calcium as gas
	950	Clay minerals converted to different phases
	600	Maximum loss of potassium and phosphorus
	540	Little residual nitrogen or carbon left
	420	Water lost from within clay minerals causing change in type
	400	Organic matter carbonized
Chemical	300	Maximum amino acid nitrogen released Loss of sulphur and phosphorous begins Organic matter charred
	200	Water repellence caused by distillation of volatiles Loss of nitrogen commences
	125	Soil sterilization
	70	High nitrate mineralization
Biological	50	Mild sterilisation owing to water loss
	<25	Usual soil temperatures

Figure 1 Changes in soil and plant material after heating (from McKenzie 2003 [26])

	Sierra*	**Escort**	**Renault**
Wheelarches	285	n/a**	725
Footwells	226	476	810

Figure 8 Table summarising maximum temperature (^oC) reached during experimentation in each location for each vehicle.

* see Figure 9 for detailed thermograph
**The wheel arches of the Escort were unaffected by the flames due to the seat of the fire being in the rear interior of the vehicle.

	Sample	Experimental conditions	Grain modification (%)
Initial Experiments	Pegmatitic quartz	900^oC for 30minutes	0
	Metamorphic schist	900^oC for 30minutes	4
	Aeolian derived quartz	900^oC for 30minutes	0
	Marine derived quartz	900^oC for 30minutes	70
	Pegmatitic quartz	1200^oC for 30minutes	4
	Metamorphic schist	1200^oC for 30minutes	100
	Aeolian derived quartz	1200^oC for 30minutes	100
	Marine derived quartz	1200^oC for 30minutes	100
Subsequent experiments	Pegmatitic quartz	1200^oC for 1hour	6
	Pegmatitic quartz	1200^oC for 2 hours	2
	Pegmatitic quartz	Crushed further to reduce particle size and subjected to 1200^oC for 30 minutes.	0
	Marine quartz	Washed and subjected to 900^oC for 30 minutes.	4

Figure 10 Table summarising the experiments undertaken for quartz of different modes of formation and provenance.

Temperature (^oC)	Salt present (Y/N)	Heat-induced textures present
No heat	N	none
800	N	none
900	N	none
1000	N	none
1100	N	none
No heat	Y	none
800	Y	none
900	Y	Glassy precipitation and single vesicles
1000	Y	Glassy precipitation and single vesicles
1100	Y	Glassy precipitation

Figure 11 Table summarising the results derived from experiment 6 investigating the effect of salt (NaCl) on the critical temperature for quartz grain surface texture modification.

Salt Type	**Temperature (^{o}C)**	**Surface texture features (% of grains)**			
		Glassy precipitation (snowdrifting)	**Single vesicles**	**Extensive vesicular structure**	**Cracks**
Table salt	800	0	0	0	0
	900	100	93	0	0
	1000	100	93	27	0
Sea salt	800	0	0	0	0
	900	100	73	0	0
	1000	100	87	0	0
Chilean salt	800	0	0	0	0
	900	80	0	0	13
	1000	80	0	0	13
Namibian salt	800	0	0	0	0
	900	87	0	0	0
	1000	87	0	13	0

Figure 12 Table summarising the results derived from experiment 6 investigating the effect of different salt compounds on the critical temperature for quartz grain surface texture modification.