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An Evaluation of Different Methods of Preparation of Quartz Grains for Study of their Surface Features by Scanning Electron Microscopy

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ABSTRACT

Sediments from periglacial and glacial environments were subjected to initial preparation and portions were subsequently further prepared using either the dichromate or the hydrogen peroxide method before being examined by scanning electron microscopy. Statistically significant differences did not occur in the results obtained for saamples subjected only to initial or to initial and either of the two further preparations: it is concluded that initial preparation is satisfactory and further preparation unnecessary.

KEY WORDS

Quartz grains, surface features, Electron microscopy.

INTRODUCTION

Electron microscope studies enabled Krinsley and Takashi [1-3] to show that quartz sand grains display distinctive surface features dependent upon the geomorphic processes that they have undergone. Margolis and Kennett [4], by recording twenty-two surface features that occur on quartz grains, established that different suites of features occur on grains subjected to glacial processes than on grains subjected to fluvial processes. Krinsley and Doornkamp [5] illustrated these and other morphological features that occur on quartz grains and suggested the origins of those features. Margolis and Krinsley [6], on the basis of suit characteristics, differentiated between grains deposited in subaqueous environments from those deposited under aeolian and glacial conditions, as well as those resulting from regolith weathering.

Higgs [7] examined thirty morphologinal characteristics and, on the basis of surface feature suites, differentiated between quartz grains deposited under fluviatile, deltaic, marine, aeolian, glacial, and pedological environments. uch subsequent research that seeks to differentiate between sediments on the basis of the processes responsible for their formation has considered the morphological characteristics of quartz grains in those sediments, based on criteria established by the above and other scientists [8].

THE PREPARATION OF QUARTZ GRAINS FOR MORPHOLOGICAL STUDY

Although morphological study of

quartz grains is of increasing importance in geological and geomorphological research, little attention appears to have been paid to the effects that different methods of preparation of quartz grains for study might have. While Walley and Krinsley [9] contented themselves with rinsing their samples well in distilled water to remove adhering surface mica flakes before picking-up and mounting their samples on standard stubs for electron microscopic examination, Krinsley and Doornkamp [5] and Wang et al [10] favoured more complicated methods. The purpose of this paper is to ascertain whether statistically different results are obtained if different methods of preparation are used.

Fine sediments were collected in Kazakhstan from avalanche deposits at an altitude of 2 500m in the Shukur valley, an east bank tributary of the Large Alma-Atinka River; the upper part of the front of a small rock glacier at 3 000m on the eastern flank of the Large Alma-Atinka valley dowslope of the Soviet Glacier; and an englacial shear plane exposed at 3 380m in the Central Tuyuksu Glacier near the head of the Small Alma-Atinka valley; all of which are located in the mountains of Zailiysky Alatau nera the city of Alma-Ata (Fig. 1).

The samples were conveyed to the laboratory and prepared for study under the electron microscope. All samples underwent the same initial preparation, which is described below. Each sample was then divided into three: one of the resultant samples received no further treatment, one was subjected to further preparation using the method described by krisnsley and Doornkamp [5], while the third was subjected to the method described by Wang et al [10].

INITIAL PREPARATION

Within the laboratory, prior to subdivision, each sample was:

i) dried at 56° C for 48 hours;
ii) weighed and divided into lots of between 10 and 11 g;
iii) digested with h2o2 in order to remove organics;

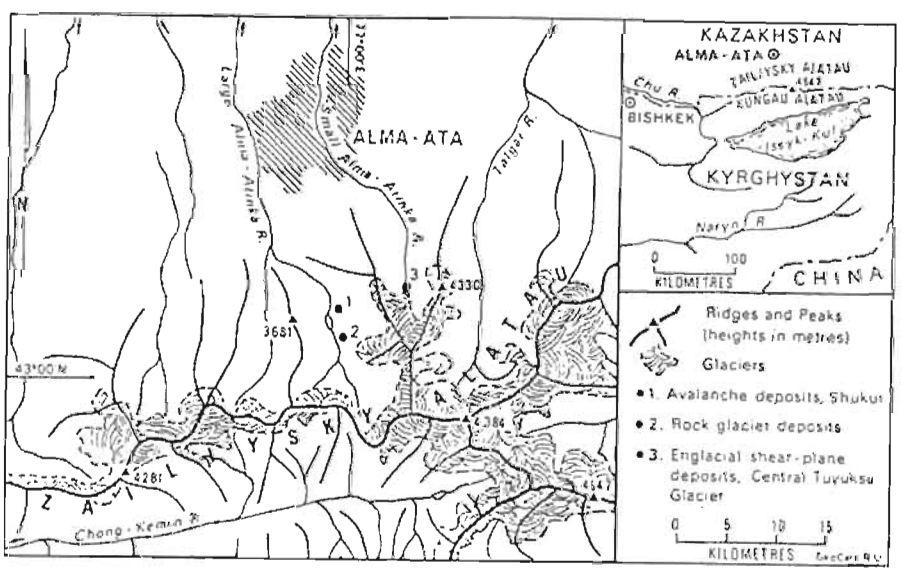

Fig. 1: The location of sample sites in Zailiysky Alatau, Kazakhstan.

iv) dried at 56°C for 48 hours;
v) reweighed;
vi) stood in a calgon solution overnight in order
to dissociate the particles;
vii) washed through a stack of sieves with mesh of 4mm, 2mm, 1mm, 0.5mm, 0.125mm and 0.063mm; the sediments from each sieve was collected and dried for up to one week at 56°C in order to evaporate water.

Each of the initially prepared samples was divided to produce three samples of sediment, each weighing about 1g, in the 0.5mm to 0.25mm size range. One sample from each set was retained for examination without further preparation; one was further prepared using the dichromate method of Krinsley and Doornkamp [5]; the other was subjected to the hydrogen peroxide method of Wang et al [10].

The Dichromate Method

Samples are placed in concentrated hydrochloric acid, boiled for ten minutes, washed thoroughly with distilled water and boiled in a stannous chloride solution for twenty minutes. Organic debris are removed in a strong oxidising solution of 1.15g each of potassium dichromate and potassium permanganate dissolved in 15 ml of concentrated sulphric acid [5]. Each sample is subsequently washed with distilled water and dried.

The Hydrogen Peroxide (H_2O_2) Method

Sample are boiled in concetrated hydrochloric acid for 10 minutes, washed with distilled water, boiled in stannous chloride for 20 minutes, then rewashed with distilled water, boiled in 30% hydrogen peroxide for 10 minutes, and finally washed with distilled water again and throughly dried [10].

Subsequent to the process of preparation used (i.e. one set of samples subjected only initial preparation, one set to initial preparation and the dichromate method, one set to initial preparation and the hydrogen peroxide method), grains were mounted with adhesive on scanning electron microscope stubs and coated with gold.

ANALYSIS OF THE SURFACE FEATURES OF QUARTZ SAND GRAINS

The surface morphology of quartz grains in each sample, prepared as outlined above, was examined, and morphological features were identified by reference to Krinsley and Doornkamp's photographic atlas of quatrz grain features [5] and Higg's [7] photographs (Fig. 2). Features were recorded using a modified version of Higg's classification as presented by Trewin [11]. Ten quartz grain prepared using only the initial preparation, ten using also the dichromate method and ten using the initial preparation and the hydrogen peroxide method were studied from both the englacial shear plane sediments and the rock glacier sediments. In the case of the avalanche debris, because of the paucity of identifiable quartz grains, (due, presumably, to the nature of the source rock) although ten quartz grains were studied from the initial preparation and ten from the initial preparation and hydrogen peroxide treated sediments, only seven quartz grain were studied from the initial preperation and dichromate treated sediments (Table1).

The percentages recorded on Table 1 were examined by means of a computer programe (STATGRAPHICS 4.0) for one-way analysis of variance. The perentages of grains from all the three depositonary environments, prepaared for analysis by each of the three methods described, and displaying the same feature, were compared wiht each other: Group

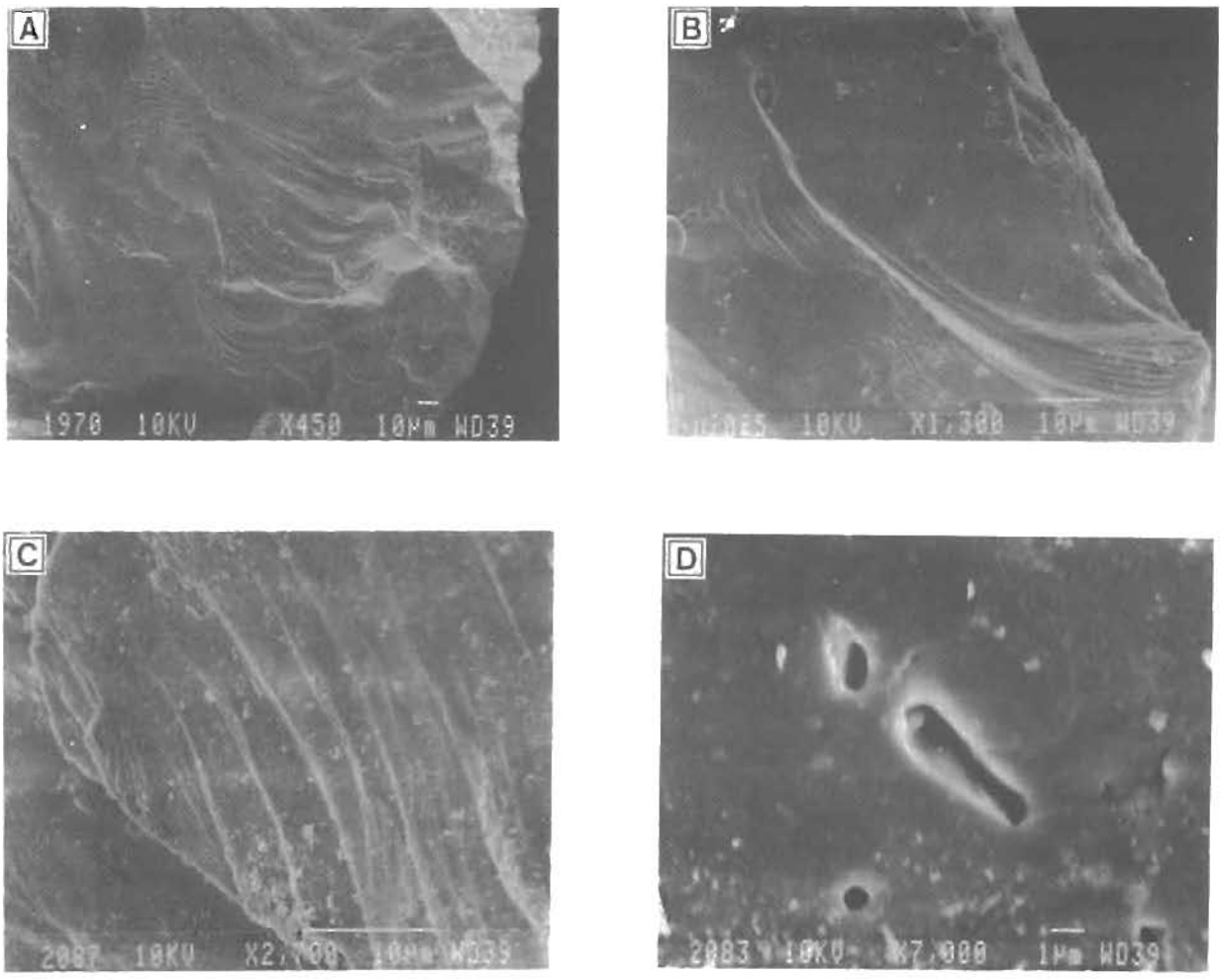

Fig. 2: Examples of morphological features identified by SEM study of quartz grains from glacial and periglacial environments in Kazakhstan.

A: arcuate steps at the base of a conchoidal fracture, avalanche debris
B: conchoidal fracture and solution pits, englacial shear plane debris
C: straight steps, englacial shear plane debris
D: solution pits, englacial shear plane debris

Feature	GROUP A			GROUP B			GROUP C		
	% of grains subjected to initial preparation on which the feature was recorded			% of grains subjected to initial and dichromate preparation on which the feature was recorded			% of grains subjected to initial and hydrogen peroxide preparation on which the feature was recorded		
	Englacial shear plane	Rock glacier	Avalanche debris	Englacial shear plane	Rock glacier	Avalanche* debris	Englacial shear plane	Rock glacier	Avalanche debris
Conchoidal Fractures	70	90	60	50	30	71	70	50	80
Steps	100	100	100	100	100	85	90	100	80
Fracture plates	100	100	100	90	50	43	10	70	70
Parallel striations	30	10	0	20	0	0	0	0	0
Imbricate grinding features	30	10	0	0	0	0	0	0	0
Adhering particles	90	100	100	100	100	100	80	100	100
Cracks	100	80	80	100	60	71	30	90	90
Scratches	60	80	100	60	90	29	20	100	0
V's	20	0	0	10	0	0	0	0	0
Solution pits/crevasses	80	50	80	100	70	85	70	70	90
Scaling	10	0	0	30	40	43	10	20	40
Crystalline overgrowths	40	90	80	70	90	29	80	100	60

* total of seven grains only

TABLE1 Morphological features identified on quartz grains, prepared for scanning electron microscope study by three different methods, from englacial shear plane; rock glacier; and avalanche debris in Kazahkstan.

A was compared with Group B and Group C, Group B was also compared with Group C. Appreciable variation of individual features occurred between groups, as with the percentage of grains collected from an englAcial shear plane that display cracks. Nevertheless, when information was aggregated into three groups as described above and the statistially analysed, no significant difference was discovered (at the 95% confidence level) between the results of the three methods of preparation, except in the case of scaling. For scaling, significant difference existed between grains subjected to initial preparation and those subjected to initial and dichromate preparation.

CONCLUSION

The three methods of preparation described produce overall results that, with the exception of scaling, were not significantly different statistically. All three methods are therefore acceptable, although it is concluded that samples only need to be subjected to initial preparation, as described in this paper.

Further preparation is time consuming, involves the use of hazardous chemicals, and there is no statistical evidence (except for scaling) that it produces significantly dofferent, or better, results.

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RESUMEN

Sedimentos de ambientes glaciales y periglaciales se sometieron a preparación inicial y subsecuentemente, partes de los sedimentos fueron tratadas por el método de dicromato o peróxido de hidrógeno, previamente a su observación por medio de microscopía electrónica de barrido. No se observaron diferencias significativas en los resultados obtenidos entre las muestras con solo la preparación inicial y aquellas en las que se aplicaron alguna de las preparaciones posteriores. Se concluye que la preparación inicial de las muestras es satisfactoria y posteriores preparaciones son innecesarias.

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