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THE EXAMINING QUARTZ GRAINS OF SEDIMENTARY ROCKS BY SCANNING ELECTRON MICROSCOPE

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Abstract. In the Carpathian Basin in the lowland areas of Hungary sediments have been accumulated in significant thickness since the Late Tertiary. Studying the material of the strata exploring deep drillings, many new data were gained about the geological development of the area. Examining the surface of quartz grains 0.63–1.0 mm in diameter taken from the sandy strata of the sedimentary rocks by electron microscope enables separating wind blown sand from that deposited by the water. Different roundedness types were defined based on detailed SEM images and determining the transporting media. On the ground of obtained results, the Quaternary development may be further specified.
The main characteristic of the deep drillings is that aeolian strata are embedded into fluvial sediments at several places. The investigations undoubtedly prove that wind-blown sand was formed several times before the Upper Pleniglacial in the Quaternary. The sand was reworked for several times in the difficult course of alluvial fan development in several places where the surface was dried.
References 12, Figs 11. In English, summary in Lithuanian.

Keywords: aeolian, fluvial sedimentary rocks, electron microscope images, roundedness of quartz grains.

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Introduction

A new drainage pattern was formed after the filling of the Pannonian Sea in the Carpathian Basin. Rivers arriving from the surrounding mountains filled the areas lying deeper in the basin by building alluvial fans and forming sediment series of significant thickness. Dryer and wetter climatic periods were alternating during the formation of the alluvial fans. Due to this, not only water but wind as well participated in the development of the surface of alluvial fans.

Significant advances took place in studying the geological development of the Hungarian Great Plain in the Late Tertiary and Early Quaternary in the second half of the last century. About 10 000 shallow (up to 10 m in depth) and almost 30 deep drillings (up to 100–1 500 m) were drilled within the frame of the long term complex research programme launched by the Lowland Division of the Geological Institute of Hungary in 1964. Processing the material of the drillings in the context of sedimentology and palaeontology, many new valuable geological-developmental, palaeoclimatologi-cal and hydrogeological data were obtained. The form and thickness of the Quaternary sediments and the hydrogeological–hydrodynamical characteristics of the several dozens of sand beds lying at differ-ent depths became known in a large area.

However numerous unsolved questions still remain concerning certain details. The determination of the rate of the aeolian strata in the sand layers of the Quaternary sediments is one among them. Estimating the erosion conditions of loose sediments and sedimentary rocks involves determining the roundedness of the quartz grains.

The fluvial or aeolian origin of the sandy sediments used to be determined on the basis of sand grains erosion – primarily, by the method of Miháltz and Ungár (1954).

On the basis of data about domestic and foreign fluvial and aeolian samples Borsy Z. (1965, 1974) it can be stated that the fluvial sand may be strongly rounded (Fig. 1). Thus the method of Mihaltz is uncertain with regard to several factors considering transporting media determination. Therefore, for accurate distinguishing between fluvial and wind-blown sand it is not enough to apply classic roundedness examinations.

In the late 1970ties, we applied the method of SEM as the resolution and depth of field seemed to be excellently applicable for studying the surface of sand grains. Under photo optic microscopes at small magnification, only the circumference can be observed and it cannot be decided whether the given appearance is a result of wearing or chemical solution or chemical crusting. In other words, it is not possible to study the circumference and the fine structure of the surface of the grains at a time.

We made images on samples taken from different domestic and foreign erosion surfaces. On the SEM images depicting typical fluvial, aeolian, marine and glacier sediment grains, the details can be observed that enabled the accurate determination of the origin (the transporting media) of the studied sediments.

After the initial success, the SEM examination of the sand strata of the deep drillings in the Great Plain was started. Such investigations of the sand sediments of the drillings (Borsy, Félszerfalvi, Lóki, 1982, 1983, 1984; Borsy et al. 1985, 1987; Franyó, Lóki, 2004) significantly contributed to correcting the late Tertiary and Quaternary surface development of the Great Plain.

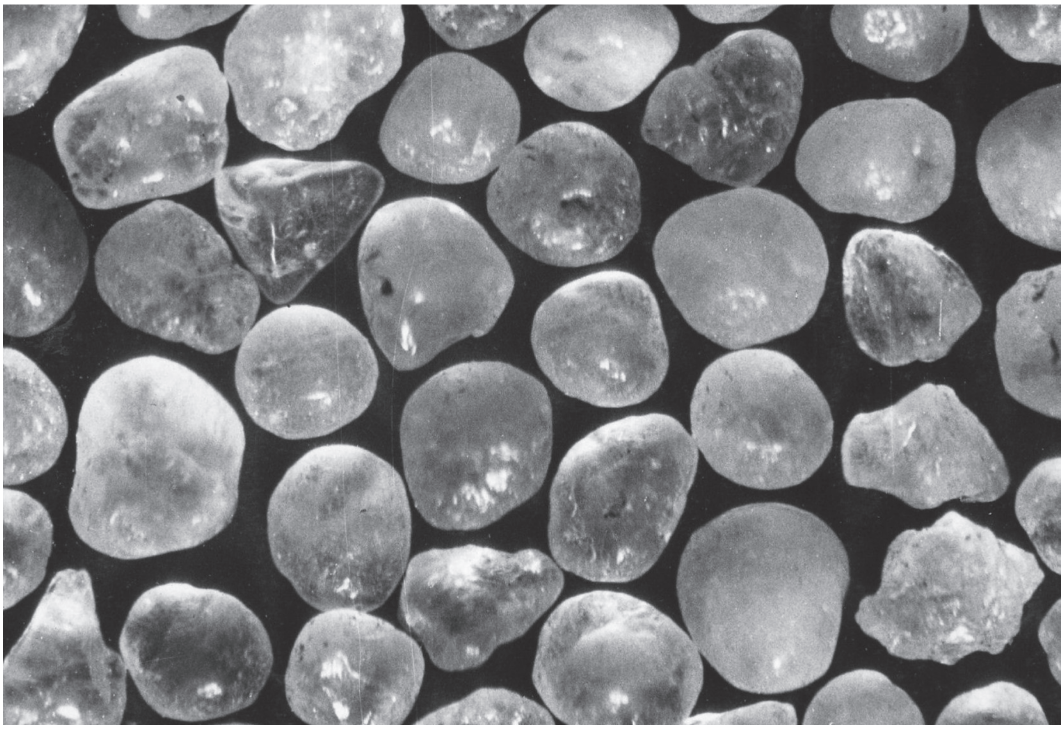

Fig. 1. Sand with a diameter of 0.63–1.0 mm of the Bug River at Zegerz (North of Warsaw)
1 pav. *Bugo upės prie Zegerco (į šiaurę nuo Varšuvos) 0,63-1,0 mm skersmens smiltelės*

1. Applied Method

Quartz grains 0.63–1.0 mm in diameter from the sand layers were used for the investigations. According to our former observations, we cannot be certain whether sands are wind-blown or fluvial based only on studying the fine fraction of the sands. Borsy Z. (1974) reported that the small and small-medium-grained wind-blown sand is dominantly saltated in the course of aeolian transport; therefore the grains are poorly rounded (Fig. 2.).

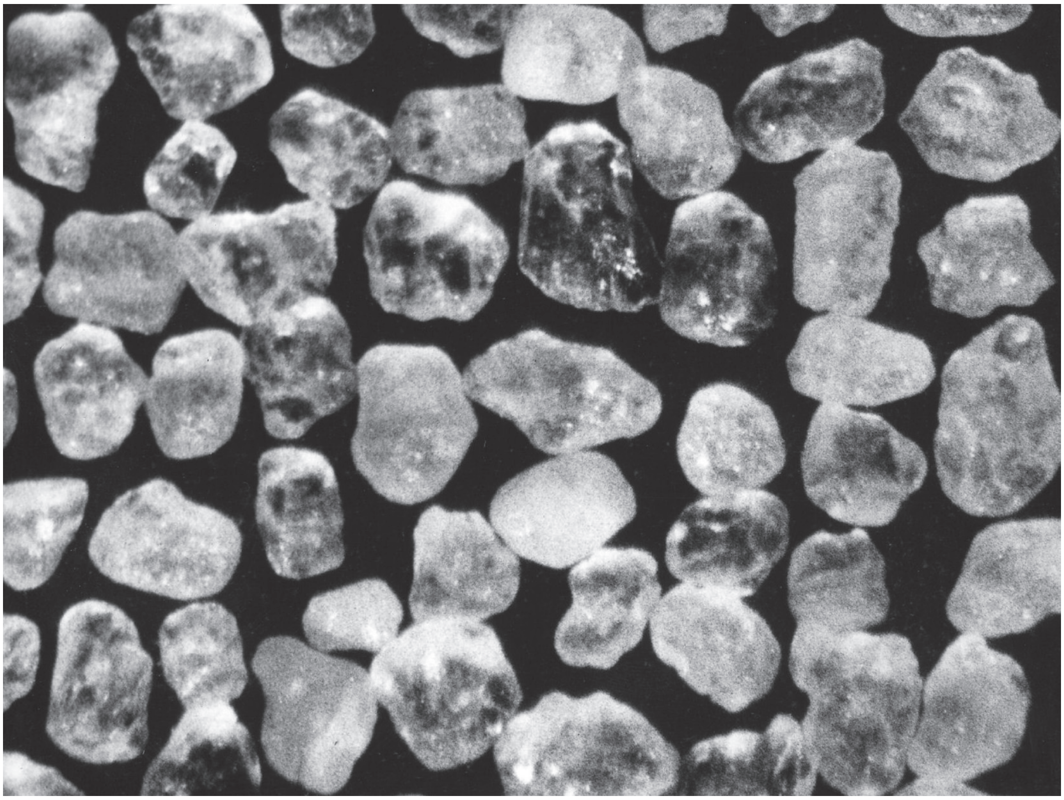

Fig. 2. Image of wind-blown sand with a diameter of 0.2-0.1 mm made by optic microscope
2 pav. *Optiniu mikroskopu gautas vėjo supustyto smėlio 0,2–0,1 mm skersmens dalelių vaizdas*

The coarse grained windblown sand, however, is rolled – primarily by the energy of the smaller grains bombing the larger ones – and saltation occurs only in very strong wind gusts. During rolling, certain marks occur on the grains within a very short time enabling us to separate the grains from the fluvial ones. In the case of fluvial transport, grains are mainly saltated thus they are poorly rounded. This is also a result of lower energy demand for grain collision in the water than in the case of wind transport.

First, the mechanical composition of the samples taken from the drillings was determined by sieving and by using Köhn pipette. Then the grains with a diameter of 0.36–1.0 mm were separated from the layers containing coarse fraction for the microscopic examination. Photos were taken from these grains both by the traditional way and by the scanning electron microscope.

For the SEM measurements, the grains were cleaned by the Krinsley and Takahashi, (1964) method. After this, the grains fixed on aluminium stumps were coated by a gold layer with a thickness of 20 nm by vacuum vaporization. Images were taken from at least 10–15 grains from each sand sam-ple for a more accurate study of the their surface. Finally, the images were interpreted on the basis of research completed at different areas (Margolis, 1968; Krinsley, Cavalerro, 1970) and of experience gained from the study of images on typical fluvial and wind-blown sands.

2. The Principles of the SEM

The scanning electron microscope (SEM) is one of the most versatile instruments for the examination and analysis of solid objects. The primary reason for its usefulness is the available high resolution (about 4 nm) and the more-or-less three-dimensional appearance of the image, which is a direct result of the large depth of field. The SEM covers a wide range of magnification (about x10 to x1 000 000) which is useful in geological and archaeological studies as well as in other fields.

The basic components of the SEM are the lens system, electron gun, visual and recording cathode ray tube, the vacuum system and the electronics associated with them. In the SEM, a finely fo-cused electron beam (one of only several nanometres) scans the specimen's surface. Different signals emit from each point of the scanning due to the interaction of the electron beam and the sample. The emitted information is then converted into an electric signal and fed into an observation screen. On the screen the information is used for controlling the contrast and the brightness of the corresponding spot. The spot on the observation screen is shown synchronously with the electron beam scanning the surface of the sample. Thus the information emitted from the specimen surface is displayed on the screen as an image (Fig. 3).

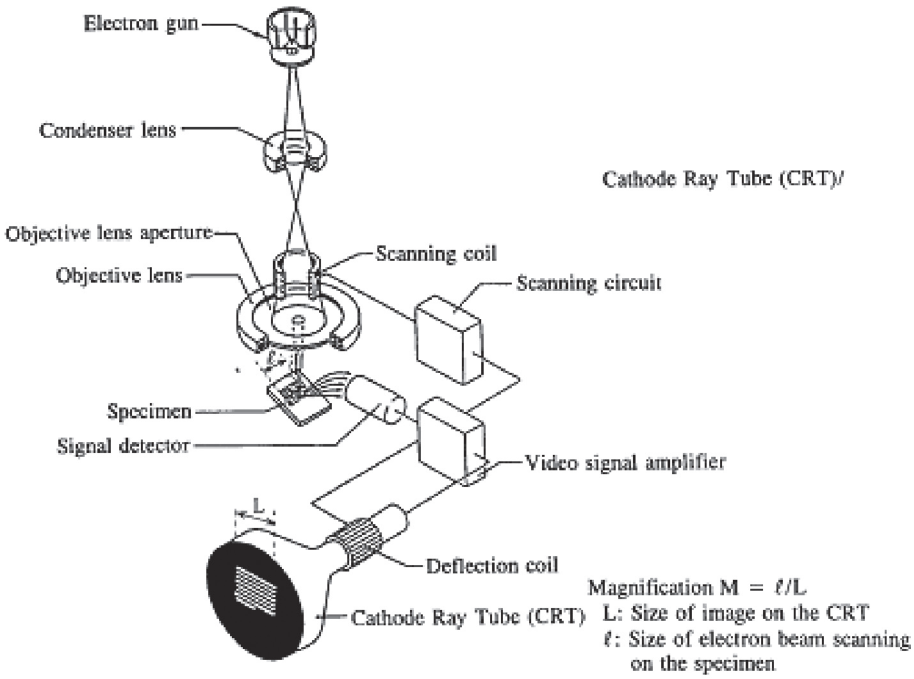

Fig. 3. Principles underlying the SEM
3 pav. *Skenuojančio elektroninio mikroskopo veikimo principas*

The type of information obtained can be various, depending on the interaction of the beam and the irradiated surface. Fig. 4. on the left shows the different type of signals obtained, while on the right it shows the regions from where the information is produced.

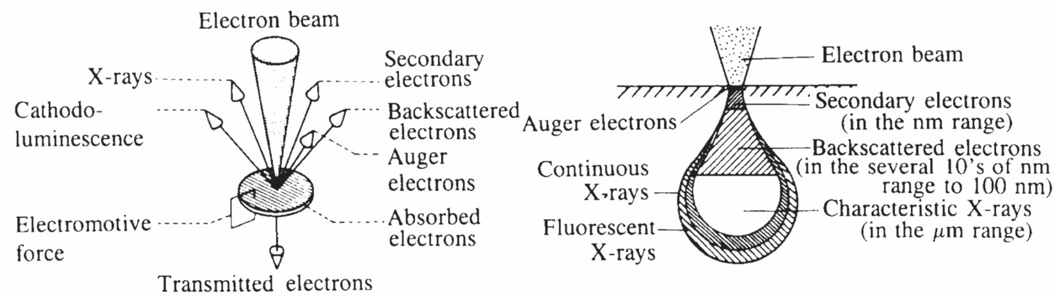

Fig. 4. Signals obtained and regions of information produced in the SEM
4 pav. *Skenuojančio elektroninio mikroskopo siunčiami signalai ir informacijos pobūdis*

The various kinds of signals carry different types of information and are used for different purposes.

The energy of the secondary electrons is very low (<50eV), thus they are emitted from the thin layer of the specimen surface meaning that the secondary electrons (SEI) can be used for topographic observations.

The energy of the backscattered electrons (BSE) is very much higher than that of the secondary electrons; they carry information from deeper layers of the surface. The number of BSE depends on the average atomic number of the sample and the incident angle of the electron beam, i.e. compositional difference can be distinguished with the backscattered electron image as well as a topographic image can be made.

Characteristic X-rays are emitted from the specimen when the electron beam irradiates it. De-tecting and analyzing the characteristic X-ray radiation, it is possible to make qualitative analysis; moreover by measuring the intensities of the characteristic X-ray lines a quantitative elemental analy-sis can be performed.

2.1. The X-ray Analysis

Thus, the addition of an X-ray analyzer to the SEM extended its capability, i.e. topographic, crystallographic and compositional information can be obtained rapidly, efficiently and simultane-ously from the same area. There are two methods to analyze characteristic X-rays; one by measuring its wave-length (wave-length dispersive X-ray spectrometry WDS) and the other by measuring its energy (energy dispersive spectrometry EDS). EDS is used more widely, because of operational ease and because it allows overall quantitative analysis to be carried out within a short time. With the EDS, X-rays are detected by an Si(Li) semiconductor detector which is separated from the vacuum system of the SEM by a thin window. The height of the current pulses generated by X-ray radiation is propor-tional to the energy of incident X-rays. Using a calibrated multi-channel analyzer, characteristic X-rays from an unknown specimen can be measured for element identification. The X-ray intensities can be converted to concentrations using one of the several methods.

2.2 Specimen Preparation

As with other microscopic techniques, the preparation of the specimen is the key of a good image. Fortunately the process which is used for optical microscopes is good enough. There is only one restriction: the sample has to be conductive. If it is not, it should be coated with conductive mate-rial, such as gold or carbon.

2.3. Experimental Technique

For the measurements an AMRAY 1830I, a scanning electron microscope was used with EDAX energy dispersive X-ray analyser. The specimens were coated with gold for imaging and with carbon for X-ray analysis. We used carbon, because the X-ray detector applied is not sensitive for light ele-ments such as C, O or N.

3. Results

During the interpretation of the images on the quartz grains separating the fluvial and aeolian grains different types were observed.

From the roundedness of the fluvial grains the distance of the erosion area can be inferred. Grains transported by water for only a small distance are worn only slightly on their edges (Fig. 5.). It is easy to interpret the fluvial transportation of strata containing such grains dominantly.

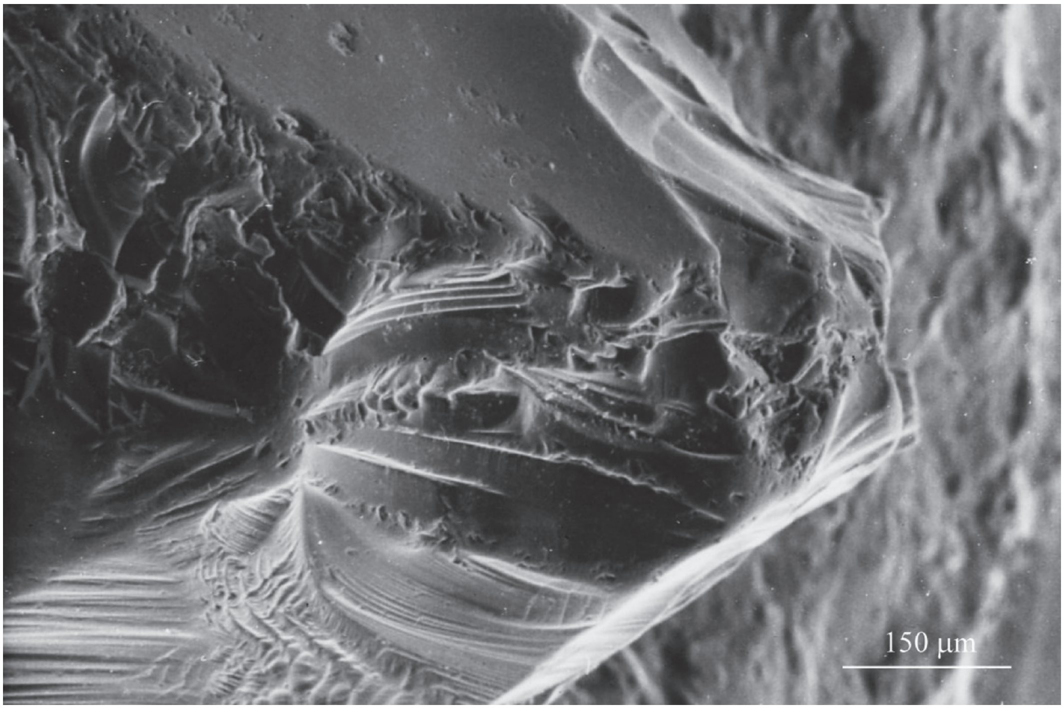

Fig. 5. Fluvial grain worn only slightly on its edges
5 pav. *Mažai apzulinta upinio smėlio dalelė*

There are sand grains worn stronger on their edges as well in fluvial strata (Fig. 6.). Differently worn grains suggest that the material of the strata originates from different regions of the catchment. Due to the effect of wearing away the breaking steps also slowly disappear.

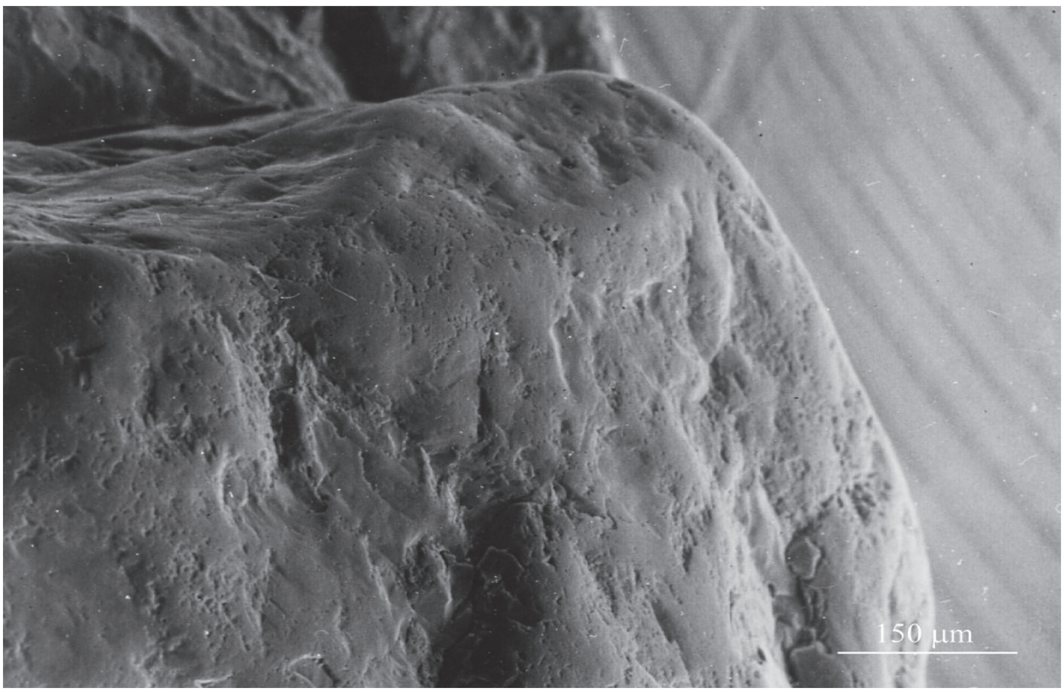

Fig. 6. Fluvial sand grain worn stronger on its edges
6 pav. *Stipriau apzulinta upinio smėlio dalelė*

On the surface of the more worn grains, the long-distance travelling depressions formed by solution also occur. It can be also observed that the edges of these depressions are also worn away due to the long water transport (Fig. 7).

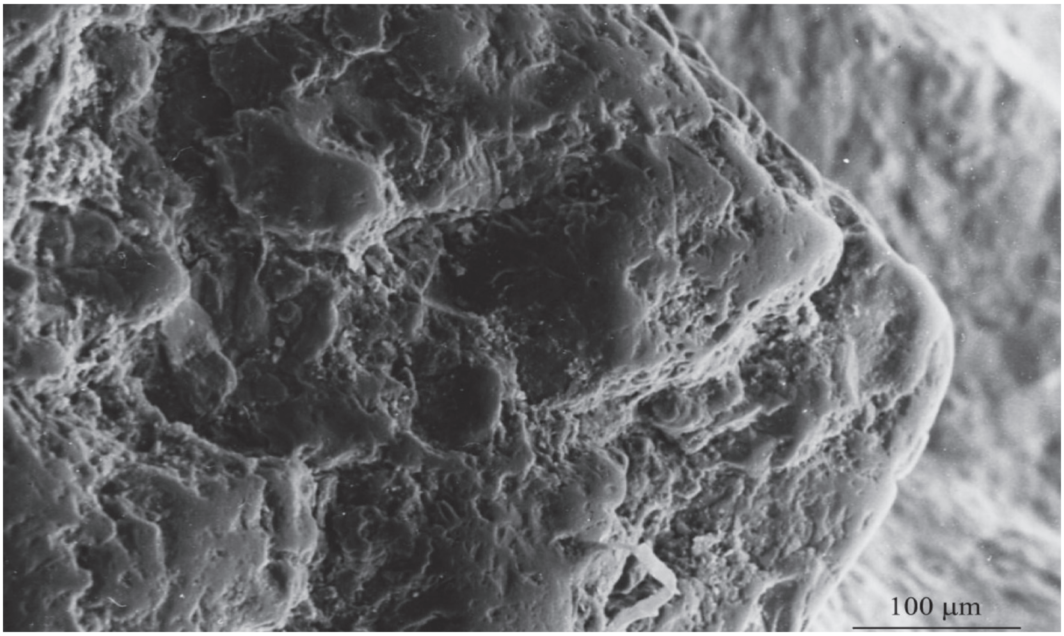

Fig. 7. Fluvial sand grain with solution marks with worn edges
7 pav. *Apzulinta ir aptirpusi upinio smėlio dalelė*

Grains from greater depth of the cored drillings show marks of strong solution (Fig. 8.). These solution marks were formed during the long time passed since the sediment was deposited. When the way of transport is to be determined on a grain like these, only those surface areas should be studied that are void of solution marks.

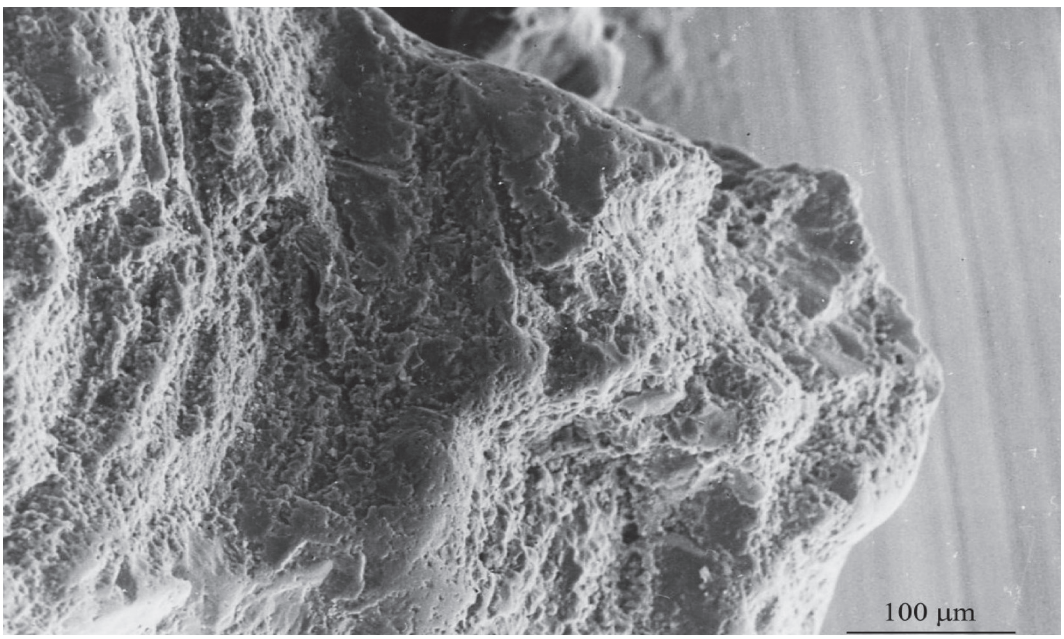

Fig. 8. Fluvial sand grain with solution marks taken from a depth of 480 m
8 pav. *Iš 480 m gylio paimto upinio smėlio aptirpusi dalelė*

Grains in shallow marine, lakeside environment are well-rounded due to the rolling back-and-forth (Fig. 9.). Drillings exposed fluvial strata containing such well-rounded grains in several places from the thick Quaternary sediment series of the Carpathian Basin. These grains were eroded and transported by the river from the sediments of shores of the Pannonian Sea/Lake.

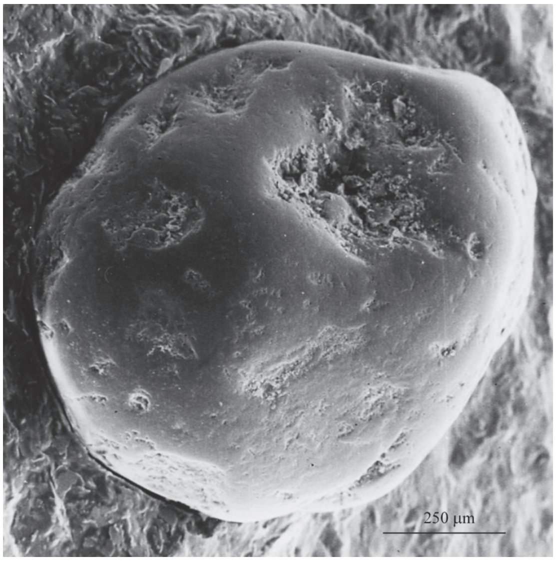

Fig. 9. Well rounded sand grain from shore environment. Solution marks are visible on the surface of the grain originating from a depth of 535 m
9 pav. *Suapvalinta priekrantės smėlio dalelė, paimta iš 535 m gylio. Matomos tirpimo žymės*

The mat surface of the wind-blown sand grains can be observed under the binocular optical microscope as well. On the SEM images at even small magnification the impacts formed by the colli-sion of the grains are well detectable (Fig. 10.). Basically this helps to separate aeolian and fluvial grains relatively easily.

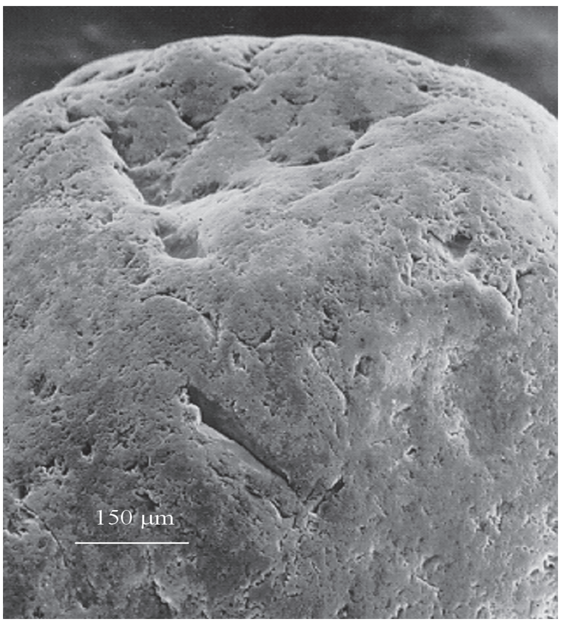

Fig. 10. Part of the surface of wind-blown sand with former solution marks
10 pav. *Vėjo supustyto smėlio aptirpusios dalelės paviršius*

Regarding roundedness, however, there are significant differences between the given wind-blown sand grains. In areas where the fluvial sand is splintery and the material blown form them is moved for a relatively short distance the roundedness of the wind-blown sand is small as well. After longer water transportation coarse sand grains become rounded as well. Therefore the roundedness of the quartz grains transported by different rivers is different and even the roundedness of the grains of the alluvial fans built by the same river may be different depending on the distance of the erosional area of the grains.

This results in the different roundedness values of grains taken from the same aeolian sample suggesting that there was significant difference in the roundedness of the sediments presenting the source of the wind-blown sand that could not have been removed by aeolian sand transport (Fig. 11.). Former solution marks can be observed on the aeolian samples as well.

The variability of the sand material of certain strata is increased by that rivers depositing the source material of the wind-blown sands sometimes eroded the area of formerly formed wind-blown sand mixing its material with the load of the river.

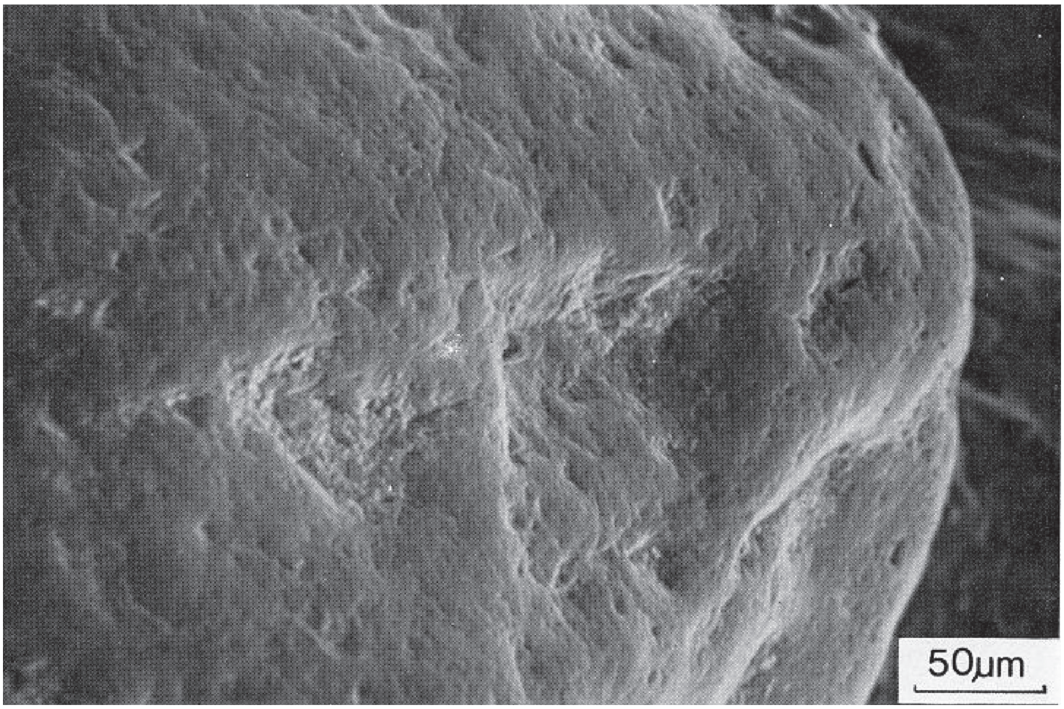

Fig. 11. Part of the surface of a wind-blown sand grains. Breaking steps characteristic of fluvial sand are still well observable at places (Jánoshalma 18)
11 pav. *Vėjo supustyto smėlio dalelės paviršiaus dalis. Vietomis gerai matyti upinėms nuosėdoms būdingos išmušimo žymės (Jánoshalma 18)*

Conclusion

Studying the surface of quartz grains taken from the sand sediments of sedimentary rocks enables determining the transporting medium. One of the main characteristics of the deep drillings is that aeolian strata are embedded into fluvial sediments. Examinations proved undoubtedly that wind-blown sand was formed several times before the Upper Plenniglacial of the Quarternary. In the difficult course of the alluvial fan formation sands were reworked several times in certain parts of the dried surface. These strata were moved into great depths at places due to tectonic movements.

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Nuosėdinių uolienų kvarco dalelių tyrimai skenuojančiu elektroniniu mikroskopu

Santrauka

Storas nuosėdų sluoksnis Vengrijos žemumose slūgsančiame Karpatų baseine kaupėsi nuo pat vėlyvojo terciaro. Daug naujų duomenų apie tiriamosios teritorijos geologinį vystymąsi buvo gauta išstudijavus nuosėdų sluoksnius gręžiniuose. Elektroniniu mikroskopu išanalizavus 0,63–1,0 mm skersmens iš smėlio sluoksnių paimtų smiltelių paviršių, atsirado galimybė skirti vėjo ir vandens suneštą smėlį. Smiltelių apzulinimo tipai buvo išskirti remiantis skenuojančiu elektroniniu mikroskopu gautais vaizdais ir nustačius pernešimo terpę. Remiantis tyrimų rezultatais galima toliau tikslinti kvartero laikotarpio bruožus.

Giliaisiais gręžiniais ištirta, kad eoliniai sluoksniai keliose vietose yra įsiterpę į upines nuosėdas. Atlikti tyrimai nepalieka vietos abejonėms, kad jau prieš viršutinįjį pleniglacialą ir kvarterą eoliniai procesai suformavo kelis smėlio sluoksnius. Ten, kur paviršius išdžiūdavo, aliuvinis smėlis iki susidarant laukams buvo ne kartą perpustytas.