TESTING OF CR-SPINEL STABILITY DURING WEATHERING, TRANSPORT, AND DIAGENESIS ON CONGLOMERATES WITH OPHIOLITIC DETRITUS: CONSEQUENCES FOR INTERPRETATION OF THE PROVENANCE OF EXOTIC FLYSCHES IN THE ALPINE-CARPATHIAN AREA

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ABSTRACT

Chrome-spinels are considered as valuable provenance indicators in heavy mineral analysis. Their importance is emphasized by the fact that they are ultrastable during weathering, transport and diagenesis. However, some earlier publications have inferred that high-Al/low-Cr spinels are less resistant which may have a strong influence on provenance studies.

In the Western Carpathians and Eastern Alps, there is a remarkable contrast between Late Jurassic to mid-Cretaceous ophiolite-bearing sediments (so-called exotics) derived from the Neotethys that are depleted in high-Al/low-Cr spinels and primary ophiolitic occurrences of Meliata Unit (also a part of Neotethys) which display the entire spectra of the Cr-spinel chemistry. It was unknown, however, whether this difference was primary or secondary, caused by later removal of high-Al/low-Cr spinels.

Therefore, Cr-spinels from the Upper Cretaceous ophiolite-bearing conglomerates in the area of the Dobšiná Ice Cave derived from the Meliata Unit were analyzed to test their stability during weathering, transport and diagenesis. Parallel analysis of spinels from sandy and pebble material showed that the spinels from both fractions cover all the chemistry spectrum from high-Cr/low-Al to low-Cr/high-Al types, without any depletion.

Good preservation of all sorts of Cr-spinels in the sandy material indicates that the depletion of Cr-spinel assemblages that was recorded in the Late Jurassic to mid-Cretaceous exotics all over the Alpine-Carpathian area appears to be primary. This was caused by lack of lherzolitic (MORB) ophiolites in the source area. Although the Meliata unit had formed a part of the same Neotethyan oceanic system, the analyzed ophiolites which include lherzolitic (MORB) types could not have been the source of Cr-spinels for the exotics-bearing flysches.

INTRODUCTION

Chrome-spinels are considered to be important provenance indicators in heavy-mineral studies (Dick and Bullen, 1983; Pober and Faupl, 1988; Lenaz et al., 2000; Kamenetsky et al., 2001). They are considered highly valuable in provenance studies due to their remarkable stability during weathering (Morton, 1984; Morton and Hallsworth, 1999, 2007; Garzanti and Andò, 2007).

The Alpine history of the Western Carpathians was influenced by two oceans (see Plašienka, 2018 for the overview) - the Neotethys (Meliata) and Alpine Atlantic (Penninic). Even though the Alpine Atlantic ophiolites are very scarce, there are two ophiolitic zones in the Western Carpathians of Neotethyan provenance. In the innermost zones there are dismembered primary ophiolites of the Meliata Unit dispersed either around or on the Gemer Unit (Fig. 1A). In the northern zones of the Internides (Tatric and Fatric units within the Core Mountains zone) and in the Pieniny Klippen Belt which is a part of the West-Carpathian Externides there are Cretaceous flysches that contain resedimented ophiolitic material in the form of pebbles to the sand grain size (especially chrome-spinels). This ophiolitic material, along with other detritic material of unknown provenance is termed exotics. The Neotethyan affinity of these exotic ophiolites has been proved previously by Dal Piaz et al. (1995) whose dating of glaucophanitic pebbles showed a Jurassic age of metamorphism.

Nearly all of the above mentioned exotics are depleted in low-Cr/high-Al spinels derived from lherzolites/MORB ophiolites (Jablonský et al., 2001; Bellová et al., 2018; Aubrecht et al., 2020, 2021). These spinels, together with those from depleted assemblages in the Upper Jurassic exotics of the Eastern Alps and Dinarides have been interpreted as being derived from harzburgitic/supra-subduction-zone ophiolites, which were obducted easier (Gawlick et al., 2015, 2020; Aubrecht et al., 2020). This, however, contrasts with Cr-spinel analyses from remnants of primary Meliatic (Triassic) and Penninic (Jurassic) ophiolites in the Alpine-Carpathian region, which also include low-Cr, high-Al spinels (Mikuš and Spišiak, 2007). The low Cr/high-Al detritic spinels appear later, in the Campanian of the Gosau Group and its equivalents (Pober and Faupl, 1988; Stern and Wagreich, 2013) as well as in the Paleogene sediments (Lenaz et al., 2009). They are mostly interpreted as being derived from northern Penninic ophiolites. Lack of this kind of detritic spinels in the earlier sediments is still unexplained.

There are some cases described in literature which showed that the provenance diagrams for Cr-spinels may not necessarily be valid for all sources. Power et al. (2000) introduced a case from the Rum layered intrusion in the Inner Hebrides, Scotland, where they revealed a wide spectrum of spinels from chromite seams, covering the entire chemistry range. In contrast, they recorded a strong shift of spinel chemistry towards Cr- and Fe-enrichment, versus Al-depletion in the grains separated from sediments of the streams draining

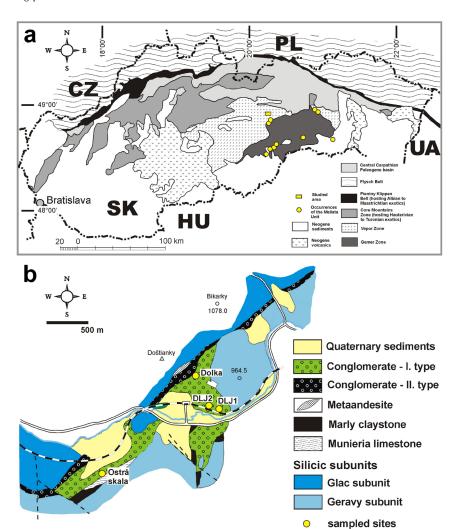


Fig. 1 - a – Position of the studied area within the schematic geological map of the Western Carpathians. b – Schematic geological map of the studied area surrounding the Dobšiná Ice Cave.

the intrusion body. This would infer a difference in stability of various Cr-spinels during weathering, transport, and diagenesis and would make the utilization of the Cr-spinels in provenance studies doubtful. Although the interpretation of Power et al. (2000) was not widely accepted by authors dealing with heavy mineral provenance, it has more than 40 SCI citations and there has been no article that has tried to disprove it to date. Therefore, doubts in Cr-spinel provenance continue to persist. The aim of this paper is to verify that the lack of low Cr/high Al spinels in the exotics is primary by performing a test of stability of Cr-spinels, based on a study of the Upper Cretaceous ophiolite-bearing conglomerates from the vicinity of the Dobšiná Ice Cave.

GEOLOGICAL SETTING

The analyzed conglomerates (Fig. 1) represent post-tectonic sediments comparable to those of the Gosau Group in the Eastern Alps, but are located in the Inner Western Carpathians and are of Neotethyan provenance, as evidenced by the presence of Triassic radiolarite clasts (Hovorka et al., 1990). At present, their age is considered Late Cretaceous (Andrusov and Snopková, 1976; Samuel, 1977), although a few earlier researchers have reported findings of Paleogene microfauna (Scheibnerová, 1960). The conglomerates contain mixture of 1) limestone pebbles from the Silicic units (Triassic limesto-

nes) which surround the basin in which they were deposited and 2) pebbles of magmatic rocks and deep oceanic sediments from ophiolitic sources interpreted as the Meliata Unit (Ivan, 1989; Hovorka et al., 1990; Ivan, 2002a; Plašienka et al., 2016), i.e., a Triassic-Jurassic oceanic domain which was a part of the Neotethys (Gawlick and Missoni, 2019).

Here, we provide a brief summary of the recent knowledge about the Meliata Unit to elucidate its lithological inventory. The Meliata Unit represents erosional remnants of a larger ophiolitic body that was formed from an accretionary wedge. Therefore, all the oceanic ophiolitic relics are dismembered and form blocks in a Jurassic sedimentary mélange. Apart from the serpentinized ultramafics they were derived from the upper part of the oceanic crust (Ivan, 2002a). The ultramafics were tectonically emplaced from the uppermost mantle as rootless bodies of various size, slivers locally reaching several kilometers in size (e.g. Komárovce serpentinite body buried under Neogene sediments), or eventually as a mélange matrix in a subduction channel (Hovorka et al, 1985). The nappe thrust of tens of kilometers was enabled by a spatial association of the Meliatic rocks with overthrusting Silicic Permian evaporites (Perkupa, or Haselgebirge Formation. - cf. Schorn et al., 2013). Currently, Meliatic remnants are preserved as isolated formations (e.g., Jaklovce, Bôrka Nappe, Meliata s.s., Bodva Valley, and Darnó formations), which differ in their original positions within the oceanic realm as well as in their subsequent metamorphic evolution (Ivan, 2002a).

The metamorphic overprint varies from rocks with preserved HP/LT subduction metamorphism, with HP/LT to LP/ LT retrogression, and initial HP/LT only in veinlets and nearly non-metamorphosed or with only a prehnite-pumpellyite metamorphic facies respectively (Arkai et al., 2003; Faryad, and Henjes-Kunst, 1997; Ivan et al., 2009; Faryad and Frank, 2011; Nemec et al., 2020). The Hačava Formation of the Bôrka Nappe contains HP/LT metamorphosed intraoceanic arc basalts (IAT) and the BABB supra-subduction type with metacarbonates and metamarls, along with serpentinitic bodies. In the Steinberg Formation of the Bôrka Nappe, there are also types close to N-MORB and metaradiolarites showing HP/LT to LP/LT retrograde metamorphism (Ivan and Kronome, 1996; Ivan, 2002b). The Jaklovce Formation contains low-metamorphosed N-MORB basalts, which are associated with radiolarites. They show evidence of initial stages of subduction metamorphism (e.g. veinlets with magnesioriebeckite) and are accompanied by serpentinized ultramafic bodies (Ivan et al., 2009). The most typical N-MORB occur in the Meliata Formation s.s. and are non-metamorphosed or just slightly metamorphosed. They are also accompanied by serpentinite bodies. In the Bodva Valley, ophiolites (mostly covered), basalts, and gabbrodolerites occur with N-MORB but also an E-MORB geochemical pattern, which are associated with radiolarites and carbonates with indications of HP/ LT to LP/LT retrogression. In the Darnó area, E-MORB-type metabasalts with carbonates and radiolarites occur, displaying signs of submarine metamorphism in LP/LT conditions. Blocks of the E-MORB-type metabasalts were likely related to the initial rift-related opening of the oceanic basin (Harangi et al., 1996; Horváth, 2000; Horváth and Árkai, 2005; Kiss et al., 2010, 2012).

Variability of geodynamic conditions of the basalt origin in various parts of the Meliata Unit points to supra-subduction zone, but also to conditions typical in oceanic rifts. Since they are always accompanied by ultramafic bodies, the presence of lherzolites, along with harzburgites is expected. In particular, the presence of a lherzolite has been confirmed at the Dobšiná location.

Paleontological data show that the age of the Meliatic oceanic rocks is Triassic-Jurassic; the sedimentary mélange matrix is Middle Jurassic (Dumitrica and Mello, 1982; Mock et al., 1998; Aubrecht et al., 2010). The age of the subduction metamorphism was determined to be within the range of 150-170 Ma (Faryad and Henjes-Kunst, 1997; Dallmeyer et al., 2008; Faryad and Frank, 2011; Putiš et al., 2019, 2022).

Although in a secondary position, the ophiolitic detritus at the Dobšiná Ice Cave represents the most complete ophiolitic suite derived from the Meliata Unit (Ivan, 2002a), reflecting the considerable lithological variability of its source. This is also supported by chemical variability of the analyzed Crspinels from the sandy matrix of the conglomerates (Mikuš and Spišiak, 2007; Sýkora et al., 2007). This variability is the largest one recorded in the sedimentary rocks all over the Western Carpathians. Therefore, they were selected as a testing material of the Cr-spinel stability during weathering, transport, and diagenesis. The testing is based on comparison of Cr-spinel assemblages from the sandstone matrix with those from the pebbles where they are still embedded in rocks. The conglomerates with prevailing limestone material (I. type conglomerate in Fig. 1) cover most of the basin, whereas those with prevailing ophiolitic material (II. type conglomerate in Fig. 1) are concentrated at the northwestern rim of the basin. The ophiolitic material consists of serpentinized peridotites, actinolitized pyroxenites, gabbros, basalts, and

deep-sea silicic sediments (Hovorka et al., 1990; Plašienka et al., 2016). Although the magmatic rocks were mostly metamorphosed and altered, their original petrographic character is still highly recognizable. Traces of intense tropical weathering during the conglomerate formation (an abundance of sesquioxides in the conglomerate matrix, impregnation of many pebbles by red iron oxides, opal clasts) are generally widespread.

MATERIAL AND METHODS

The sample material consisted of four samples of more than 5 kg of conglomerates per sample, containing a sufficient amount of ophiolitic material. Sample DLJ1 was taken at the railway undercrossing north of the Dobšiná Ice Cave (N 48°52'30.7", E 20°18'11.8", Fig. 2b). Sample DLJ2 was taken west of the previous sample at the local road running parallel to the railway (Fig. 2a), near the local train stop (N 48°52'32.1", E 20°18'02.6"). Sample Ostrá skala (Fig. 2c) was taken from the conglomerates in a forest road SW of the Ostrá Skala cliff west of the Dobšiná Ice Cave (N 48°52'04.84'', E 20°16'53.95''). Sample Dolka (Fig. 2d) was taken from a forest road-cut north of the Dolka settlement and north of the Dobšiná Ice Cave (N 48°52'45.8", E 20°17′54.2′′). The samples of conglomerates were dissolved in acetic acid. Pebbles were picked by hand from the residuum (Fig. 2d). Most of the weathered ophiolitic magmatics turned red, while the rest attained a greenish colour. The red coloured magmatic pebbles could not be discerned from those of red radiolarites; therefore they were extracted together (radiolarite pebbles do not contain Cr-spinels and thus could not influence the final results). The sand fraction (0.07-1 mm) was sieved and heavy minerals were extracted by heavy liquid (sodium polytungstate). The pebbles (larger than 0.5 cm) were crushed (to about 2.5 mm in order to avoid spinel grain breakage) and the heavy minerals derived from them were separated similarly. The heavy fractions resulting from the process were all mounted into epoxide resin, polished and analyzed in a microanalyzer. Using whole heavy fractions instead of picking Cr-spinels manually prevented random avoiding of some types of spinels.

Chemical compositions of the spinels were analyzed using a JEOL JXA-8530FE microprobe (Earth Science Institute of the Slovak Academy of Sciences in Banská Bystrica, Slovakia). Analytical condition were as follows: accelerating voltage 15kV, sample current 20nA, probe diameter 2-10 μ m, counting time 10s – peak and 5s for background. Raw counts were corrected using a PAP routine on CAMECA and ZAF correction were used. The standards used were: Ca - diopside, Mn – rodonite, Si - quartz, Mg – olivine, F – fluorite, Na – jadeite, Al– kyanite, K – orthoclase, Fe – hematite, Ti - rutile, Cr - Cr₂O₃, Cl - tugtupite. All measured spectral lines are $K\alpha$. Only fresh preserved magmatic cores of spinels were measured. The analyses are reported in Table 1S (Supplementary Material). The composition of the altered rims was beyond the scope of this paper and the interested readers are referred to the publication of Mikuš and Spišiak (2007).

RESULTS

To determine the provenance of spinels, it is essential to analyze the chemical variability of stable elements such as Mg, Fe, Cr, Al, and Ti.



Fig. 2 - Examples of the studied conglomerates. a – One of the classical outcrops in a roadcut near the railway station (DLJ2 sampling site) showing conglomerate with limestone-dominated pebbles. b – Ostrá skala sample showing increased content of ophiolitic pebbles (red and green). c – Part of the DLJ1 sample showing limestone-dominated pebbles. d – Part of the Dolka sample during the laboratory dissolution process. The pebbles are mostly formed by ophiolitic material. However, a relatively low number of Cr-spinels extracted from the pebbles indicates that most of the ophiolitic pebbles were radiolarites, or magmatic rocks that did not contain Cr-spinels.

Two types of diagrams were employed to interpret spinel provenance. The first one, developed by Pober and Faupl (1988), plots Mg/(Mg + Fe²⁺) against Cr/(Cr + Al) and is designed to distinguish spinels derived from harzburgites, lherzolites, podiform chromitites, and cumulates (Fig. 3). The analyzed spinel grains span the entire lherzolitic and harzburgitic fields, with some overlap into the cumulate field. Grains from the sandy fraction exhibit a relatively continuous distribution across these fields, whereas those from the pebble fraction show a more discontinuous or patchy pattern. Nevertheless, spinels from both fractions are present in all identified compositional fields.

The second diagram used for provenance analysis is the Al_2O_3 versus TiO_2 plot, introduced by Lenaz et al. (2000) and Kamenetsky et al. (2001). This diagram is based on the observation that most mantle-derived spinels contain less than 0.2 wt.% TiO_2 , whereas the majority of volcanic spinels exhibit TiO_2 concentrations above 0.2 wt.% (for a comprehensive review, see Lenaz et al., 2009). The diagram (Fig. 4) again shows that the analyzed grains cover the entire spectrum from the supra-subduction zone peridotites to the mid-oceanic

ridge peridotites. The pebble spinels from DLJ2 and Dolka samples are concentrated exclusively in the supra-subduction zone peridotites field.

The summarizing histogram (Fig. 5) of Cr/(Cr + Al) distribution of shows a relatively even distribution of the spinel chemistries of the grains from sandy fraction, as well as relatively uneven distribution of those from pebbles. However, the grains from pebbles cover the same entire spectrum as the sand grains, with maxima approximately corresponding to spinels from sandy fraction.

INTERPRETATION AND DISCUSSION

Spinel stability during weathering, transport and diagenesis

The analysis results show that there is no depletion in high-Al/low-Cr in the sand detritus. However, the spinels from pebbles show wide, but discrete, patchy distribution, which is caused by several factors. 1) Only in the Ostrá skala sample does the spinel chemistry from pebbles cover the entire spec-

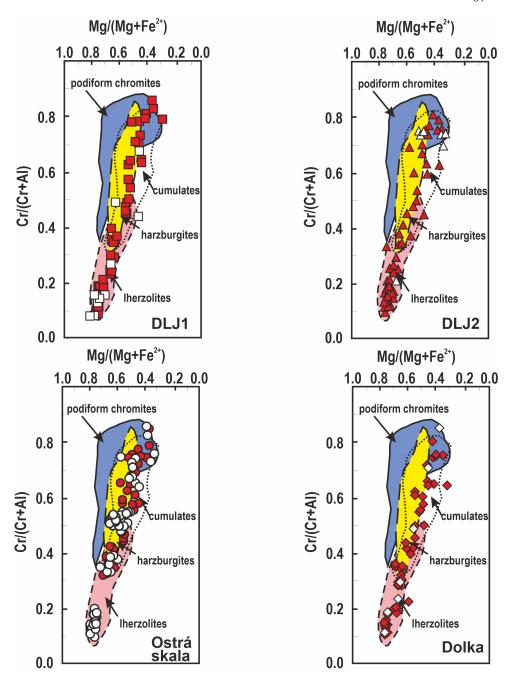


Fig. 3 - Spinel composition plotted in the $Mg/(Mg + Fe^{2+})$ vs. Cr/(Cr + Al) diagrams with fields defined according to Pober and Faupl (1988). Diagram fields: solid line – podiform chromitites, dotted line – cumulates, dashed line (short dashes) – lherzolites, dashed line (long dashes) – harzburgites. Empty symbols – spinels from pebbles, red symbols – spinels from sand fraction.

trum; most of the ophiolitic pebbles at other locations were either sedimentary, or magmatic rocks without Cr-spinels. 2) The majority of the Cr-spinel-bearing rocks were serpentinized and may have disappeared. Serpentinites have very low stability during weathering, transport and diagenesis. They are highly susceptible to alteration during weathering and transport. For instance, only tiny fragments (less than 1 mm) of serpentinite were found in the exotic conglomerates in the Klape and Kysuca units of the Pieniny Klippen Belt (Mišík and Sýkora, 1981), despite having a high content of Cr-spinels in the sandy fraction.

Nevertheless, in the example of the conglomerates from the Dobšiná Ice Cave area it is obvious that the analyzed Crspinels from the sandy fraction have not been affected by weathering and transport and they completely cover all the chemistry spectrum, including high-Al/low-Cr parts. This confirms earlier assumptions about the high stability of Crspinels of all chemistries during weathering, transport and diagenesis thereby contradicting to the results of Power et al.

(2000). Their explanation that Al is lost during serpentinization and the resulting grain chemistry is commonly shifted towards higher Cr and Fe contents has not been confirmed. All the microprobe analyses either previously in the exotic flysches or presently in the Dobšiná Ice Cave conglomerates dealt with grains that were of approximately the same size; the cores and alteration rims were not taken into account in this study. By measuring only the grain centres, an eventual complete alteration of the high-Al grains would be visible in the diagrams (Fig. 3-4) presented herein since their chemistry often plots outside of the pre-defined fields.

Was the Meliata Unit the source of the exotics?

The test results from the Dobšiná Ice Cave area show that the general lack of high-Al/low-Cr spinels in the exotic flysches can be considered primary and not influenced by a secondary removal of low-Cr/high-Al spinels. The Neotethyan oceanic crust is considered to be the main source of

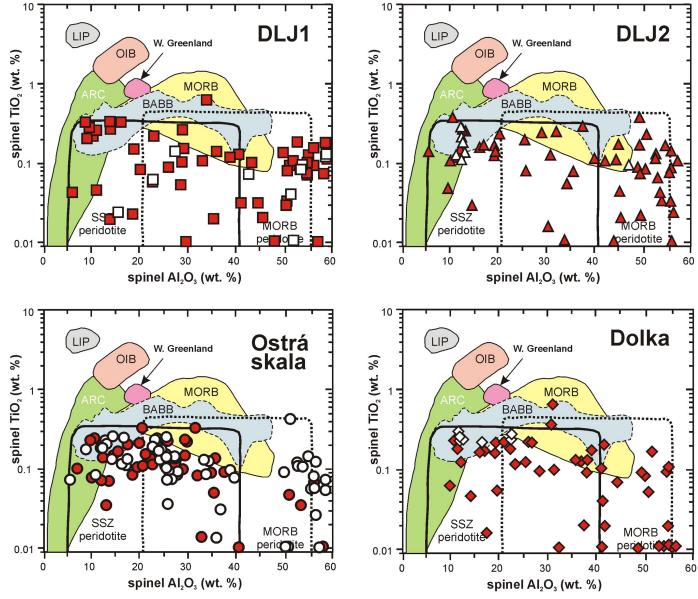
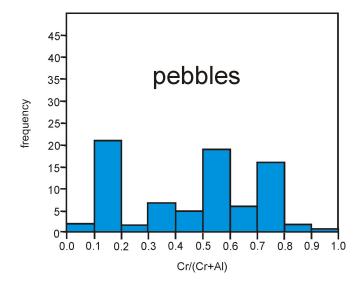


Fig. 4 - Spinel composition plotted in the TiO_2 vs. Al_2O_3 diagram of Lenaz et al. (2000) and Kamenetsky et al. (2001). Explanations: LIP - large igneous provinces, OIB - ocean island basalts, ARC - island-arc magmas, BABB - back-arc basin basalts, MORB - mid ocean ridge basalts, SSZ - supra-subduction zone peridotites. Empty symbols - spinels from pebbles, red symbols - spinels from sand fraction.

the exotic ophiolites. This has also been proven by Dal Piaz et al. (1995) and Putiš et al. (2023), who determined Late Jurassic age of metamorphism for the blueschist pebbles in the exotics. Nevertheless, the results indicate that the Meliata Unit likely originated from a different part of the Neotethys with respect to the source area of the exotic sediments. Large geochemical variability of the Meliata Unit ophiolites is underlined by a wide spectrum of Cr-spinels derived from it (Mikuš and Spišiak, 2007) and the ophiolites in the exotics are different from the tested conglomerates at the Dobšiná Ice cave. The Cr-spinel analyses show that lherzolitic (MORB) types were generally absent in the source area of the exotics. Widespread distribution of Cr-spinels in the exotic flysches was most likely caused by a large-scale obduction of oceanic crust. This generally rare process more likely occurs when upper-plate supra-subduction ophiolites are obducted, as opposed to those originating in mid-oceanic ridges which typically belong to the lower plate and are commonly destroyed by subduction. Therefore, the interpretation of obduction of SSD ophiolites dominated by harzburgites, which has been proposed in several recent studies on exotic ophiolitic material (Gawlick et al., 2015, 2020; Aubrecht et al., 2020), seems to be substantiated. The Meliata Unit was most likely transported in a different way, e.g. as dismembered ophiolites at the base of the higher overthrusting units within the accretionary wedge. Hence, they were uncovered much later than the SSD ophiolites which had been obducted since the Late Jurassic.

The depletion of Cr-spinels in Late Jurassic and mid-Cretaceous exotic sediments throughout the Alpine-Carpathian region appears to be a primary characteristic, rather than the result of secondary removal of low-Cr/high-Al spinels. This depletion is attributed to the absence of lherzolitic ("MORB-type") ophiolites in the source area, rather than selective loss during weathering and transport. This conclusion is supported by Cr-spinel analyses of Upper Cretaceous ophiolite-bearing conglomerates from the Dobšiná Ice Cave, which originated in the Meliatic (Neotethys) oceanic domain."



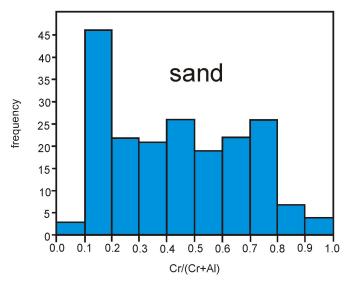


Fig. 5 - Summary histograms showing distribution of Cr/(Cr+Al) for spinels in the sand and pebble fractions.

CONCLUSIONS

- 1. The depletion of Cr-spinel assemblages in the Late Jurassic and mid-Cretaceous exotics all over the Alpine-Carpathian area appears to be primary rather than the result of secondary removal of low-Cr/high-Al spinels. This depletion is attributed to the absence of lherzolitic ("MORB-type") ophiolites in the source area, rather than selective loss during weathering and transport. This conclusion is supported by Cr-spinel analyses of Upper Cretaceous ophiolite-bearing conglomerates from the Dobšiná Ice Cave, which originated in the Meliatic (Neotethys) oceanic domain. Parallel analyses of spinels from both sandy and pebble fractions demonstrate that they span the entire compositional spectrum, ranging from high-Cr/low-Al to low-Cr/high-Al types.
- 2. The analyzed ophiolites of the Meliata Unit which include lherzolitic (MORB) types show a highly variable geochemical pattern. Although the Meliata unit formed a part of the Neotethyan oceanic system, the analyzed ophiolites could not be the source of Cr-spinels of the exotics-bearing flysches; they were derived exclusively from the back-arc, upper plate ophiolites.

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