MINERALOGICAL, PETROGRAPHIC AND GEOCHEMICAL STUDY OF NEOLITHIC POLISHED AXES FROM MICULA (NW TRANSYLVANIA, ROMANIA)

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Abstract

Two polished stone axes found at Micula (NW Transylvania, Romania) were mineralogicaly, petrographicaly and geochemicaly studied. The stone artefacts were assigned to the Neolithic period. Both are made from metamorphic rocks, i.e. amphibolite and hornfels respectively. The petrography, EMP, and PGAA analyses of the stone axes compared with geological information and the references data point to an extremely large range of possible geological sources for the rocks: from the northern part of the Apuseni Mts. to the northern part of the Eastern Carpathians or even the Bohemian Massif. Most likely, the place for collecting material could have been the alluvial sediments (boulders, pebbles) from the rivers nearby, such as Someş, Tisa, Crasna or Criş.

Kivonat

Tanulmányunkban a Szatmár-megyei Mikolából (ÉNY Erdély, Románia) származó két csiszolt kőbaltát vizsgáltuk meg, ásványtani, kőzettani és geokémia szempontból. A kőeszközök a csiszoltkőkorszakhoz sorolhatók. Mindkettő metamorf kőzetből készült, amfibolitból és szaruszirtből. A kőzettani és a prompt gamma aktivációs analízis, valamint a rendelkezésre álló földtani ismeretek alapján az amfibolit kőbalta esetében a nyersanyag származása több lehetőséget is felvet, az Erdélyi-szigethegységtől, a Keleti-Kárpátok északi vonulatán át egészen a Cseh-Masszívumig. Ugyanakkor primer nyersanyaforrásként a Szamos, Tisza, Kraszna és a Körösök folyóüledéke is valószínűsíhető.

Rezumat

Lucrarea prezintă rezultatele studiului mineralogic, petrografic şi geochimic asupra a două topoare şlefuite descoperite în raza localității Micula (NV Transilvania, România). Uneltele au fost atribuite epocii neolitice si sunt executate din roci metamorfice, unul dintr-un amfibolit, celălalt dintr-o corneeană. Caracterele petrografice şi rezultatele analizelor de microsondă electronică şi Activare Prompt Gamma comparate cu informatii geologice şi cu surse bibliografice indică o gama larga de posibile surse geologice: de la nordul Munților Apuseni la nordul Carpatilor Orientali si chiar Masivul Bohemian. Cel mai plauzibil, rocile din care au fost confecționate topoarele au fost colectate nu direct din aflorimente ci din pietrişurile aluviale ale unor râuri din regiune: Tisa, Someş, Crasna sau Criş.

KEYWORDS: NEOLITIC POLISHED STONE AXE, AMPHIBOLITE, HORNFELS, MICULA (MIKOLA), TRANSYLVANIA, ROMANIA, XRD, EMPA, PGAA

KULCSSZAVAK: NEOLIT CSISZOLT KŐESZKÖZ, AMFIBOLIT (METABÁZIT), MÉSZ-SZILIKÁT SZARUSZIRT, MIKOLA (MICULA), ERDÉLY, ROMÁNIA, XRD, EPMA, PGAA

CUVINTE CHEIE: UNELTE ŞLEFUITE NEOLITICE, AMFIBOLIT (METABAZITE), CORNEEANĂ, MICULA (MIKOLA), TRANSILVANIA, ROMÂNIA, XRD, EPMA, PGAA

Introduction and archaeological context

The paper presents the mineralogical, petrographical and geochemical data on two polished stone axes found in the surroundings of the Micula village (Hungarian name: Mikola) from the Satu Mare County, in the north-westernmost part of the Transylvania, Romania (**Fig. 1**). The tools were collected during field survey, and not upon systematic research. No ceramics fragments were found nearby. Based on the shape, it can be considered that the stone tools are most likely of Neolithic age. The tool labelled II.3 was found on the bank of the Vármegyárka rivulet, south of

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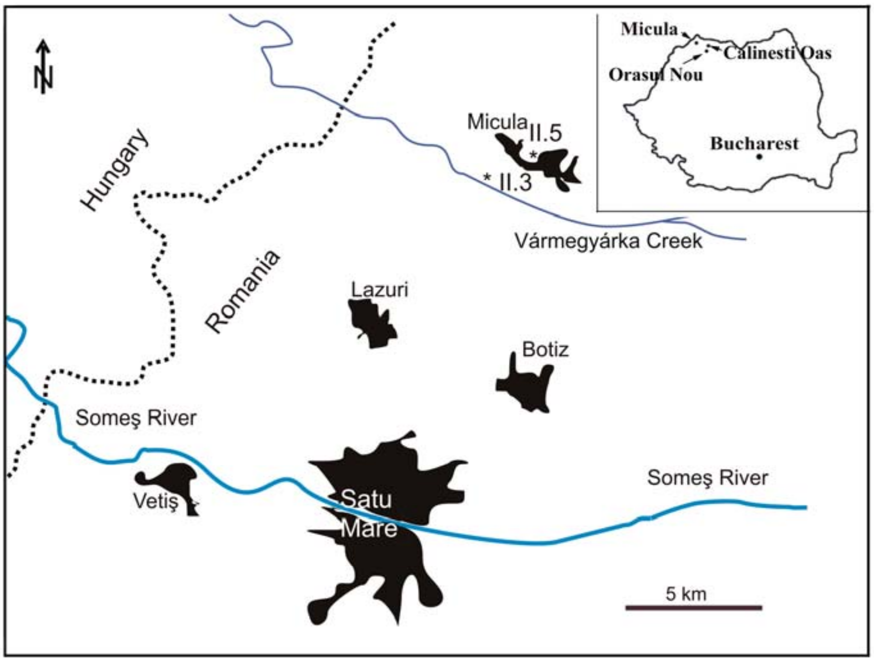

Fig. 1.

Location of the stone tools findings (II.3 and II.5) nearby Micula (Mikola) village. The right-upside insert shows the location of the village on the map of Romania

Micula village (GPS location: N 47º89`56``; E 22º92`84``). The tool labelled II.5 was found north of the village (GPS location: N 47º90`09``; E 22º95`49``).

Few archaeological studies on Neolithic Age remnats are recorded in the area, e.g. BITIRI & SOCOLAN (1966), for the Călineşti-Oaş site and SIMON et al. (2003) for the Piatra Curmeni and Oraşul Nou sites. Micula village history starts in the 13th century according to unpublished data by Sándor Gellért (1916–1987). No data on prehistoric artefacts findings in the area are available so far.

The mineralogical and petrographic characterisation of stone tools and the provenance research i.e. the finding of the possible geological sources used for raw materials represents aims of the archaeometric and geoarchaeological studies. Upon analysing prehistoric tools we can get an insight into the type of raw materials which were used. Additionally, answers to the questions such as why the craftsmen preferred a certain lithic material and how the tools were shaped are other archaeological outcomes. By combining the mineralogical-petrographical and geochemical data with the archaeological background, new social, technological and economical information, including the trace of trading routes of an ancient society may result. The aim of this paper is to define the petrographic type of the stone tools and to compare it with known geological sources.

Samples and methods

The stone tools were labelled as: II.3 and II.5. Two thin slices were cut from each tool with an ISOMET 11-1180 LOW SPEED SAW type (BUEHLER Ltd.) cutter. The slices were used for thin sections for optical microscopy (OM) in plane-polarized light.

For mineral identification, few grams taken from the axe II.3 were analysed by X-Ray Powder Diffraction (PXRD). The Siemens D500 diffractometer[1] worked at room temperature, at 41 kV and 20 mA, with CuK_α radiation (λ = 1.54178 Å), and graphite monochromator. The scan speed was 0.02°/step, from 2 to 75° 2θ.

Electron microprobe analyses (EMPA) on polished thin sections coated with carbon were carried out at 15 kV accelerating voltage, 40 nA beam current intensity and 5 μm electron-beam diameter, with a Jeol XA 8600 equipped with four wavelength dispersive spectrometers (WDS) and Si(Li) energy-dispersive spectrometer (EDS).

Scanning electon microscopy (SEM) on polished samples coated with gold were carried out at 20 keV voltage, 1–2 nA current intensity, with an AMRAY X 1830 I/T6, equipped with EDAX PV 9800 ED spectrometer[2].

Prompt Gamma Activation Analysis (PGAA) was carried out at the Institute of Isotope and Surface Chemistry Budapest. The water-cooled, water-moderated VVR-type reactor has a 10 MW thermal power. A beam of thermal neutrons is guided to 30 m away from the reactor through a device made from special glass coated with Ni. The neutron flux

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which hits the sample to be analyzed has 5–10 meV and contains 2.5 x 10^6 neutron times $cm^{-2}s^{-1}$. The sample emits gamma rays which are detected by a high-purity Ge detector (HPGe), surrounded by eight bismouth-germanate (BGO) scintillation detectors. The resulted spectra were identified with "Hypermet PC" software. The PGAA is a non-destructive (RÉVAY *et al.,* 2000; KASZTOVSZKY *et al.,* 2002; SZAKMÁNY & KASZTOVSZKY, 2004; KASZTOVSZKY *et al.,* 2008) and can identify not only major elements but trace elements as well.

Results

The hornfels axe (no. II.3)

The axe labelled II.3 has an elongated shape (10.8 x 3.9 x 2.7 cm) and is made from hornfels[3]. The colour of the sample in fresh break is greenish-gray. The surface of the tool is covered by a light brownish weathered layer, of 1 mm thickness. The elongation of the axe follows the natural schistosity of the rock, marked by thin, white bands arranged in almost parallel rows (**Fig. 2**).

The rock consists mainly of feldspar and pyroxene grains, closely associated. Few idiomorphic crystals of pyrite up to 200 microns in size, as well as some feldspar porphyroblasts were noticed. Late deposition of microquartz into elongated voids is shown in **Fig. 3.** The Back Scattered Electron (BSE) image (**Fig. 4**) reveals that the rock consists of a fine mixture of different minerals, mainly potassic feldspar, quartz and pyroxene (diopside). Besides the main minerals, other minerals such as anthophyllite – a Mg-Fe amphibole, clinochlore, apatite and carbonates may occur in small quantities. The carbonates are rich in Fe (siderite) and have a low content of Mn.

Fig. 2. - Macroscopic image of the hornfels axe (no. II.3) and the slice cut for the thin section (scale bar = 5 cm)

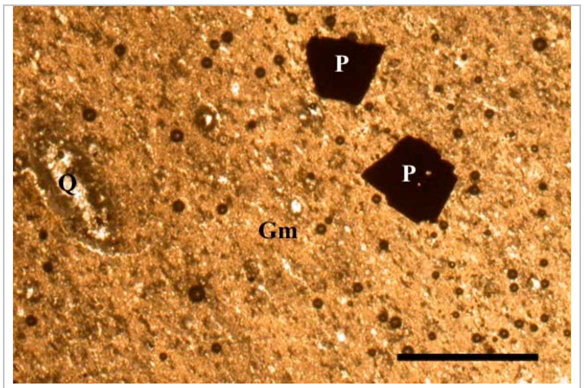

Fig. 3. - Thin section of the hornfels axe: microphoto in plane-polarized light. Abbreviations: Q for quartz, P for pyrite crystals, Gm for the groundmass composed of a fine mixture of feldspar and clinopyroxene. Crossed nicols. Scale bar = 0.5 mm

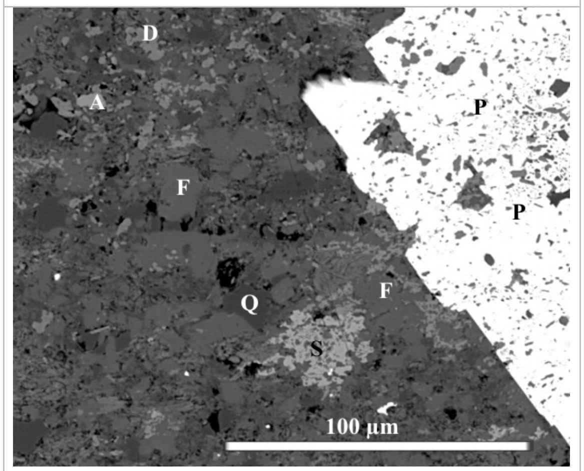

Fig. 4. - BSE image of the axe hornfels axe (II.3). Abbreviations: P – Pyrite; Q – Quartz; F – Feldspars; A – Anthophyllite; D – Diopside; S – Siderite.

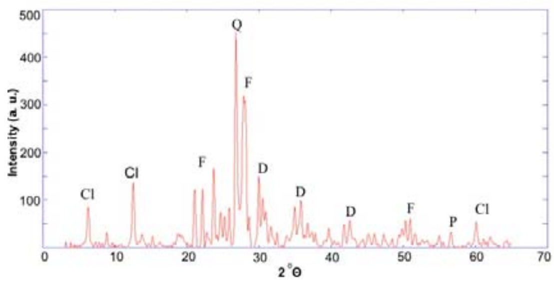

Fig. 5. - Diffractogram of the hornfels axe (no. II.3). Abbreviations: F – feldspar, Q – quartz, P – pyrite, D – diopside, Cl – Clinochlore

[3] A fine-grained, compact and tough metamorphic rock formed on the expenses of clayish sediments heated by the contact with hot magmas. Often used for stone tools in prehistory.

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Identification of the point analyses	Calculated structural formula	Mineral phase identified	Note	Related points on Fig. # 4
NV01L1	$Ca_{0.87}Mg_{0.76}Fe^{2+}_{0.33}Mn^{2+}_{0.02}Al_{0.03}[Al_{0.09}Ti_{0.01}Si_{1.90}O_6]$	*Diopside*	1	D
NV01N1	$Ca_{0.91}Mg_{0.71}Fe^{2+}_{0.29}Mn^{2+}_{0.02}Al_{0.06}[Al_{0.04}Si_{1.96}O_6]$	*Diopside*	1	-
NV01K1	$K_{0.9}Na_{0.08}Ca_{0.01}[AlSi_3O_8]$	*K-feldspar*	2	F (right side grain)
NV01P1	$K_{0.98}Ca_{0.02}[AlSi_3O_8]$	*K-feldspar*	2, 3	F (left side grain)
NV01O1	$Mg_{2.51}Fe^{2+}_{1.86}Al_{1.28}Mn^{2+}_{0.05}Ca_{0.04}[Al_{0.75}Si_{3.25}0_{10}](OH)_8$	*Clinochlore*	4	-
NV01J1	$Fe_{0.78}Ca_{0.12}Mn_{0.10}[CO_3]$	*Siderite*	5	S

Table 1. - Calculated structural formulae for minerals of the hornfels axe. All phases were identified on group level by polarized light microscopy in the thin section prior to the standardless SEM+EDX analyses. Notes: 1 – calculated for 6 oxygen; 2 – calculated for 8 oxygen; 3 – Mg 0.81 and Fe^{2+} 0.25 were omitted from the calculations as traces of admixtured other phase(s); 4 – calculated for 14 oxigen; 5 – calculated for 1 oxygen.

Table 1 displays the chemical composition of some minerals of the hornfels axe.

The X-Ray diffraction identified the presence of feldspar, diopside, clinochlore, quartz and pyrite, as displayed in **Fig. 5**.

The amphibolite axe (no. II.5)

The stone tool labelled II.5 is made from an amphibolite[4] and has a flattened rectangular shape (**Fig. 6**), with 3.0 x 2.6 x 0.9 cm size. Seen in fresh break, the colour is dark greyish-green; on the exposed and weathered surfaces, the colour is light grey, with small brownish spots.

Fig. 6. - The amphibolite axe (no. II.5) and the slice used for thin section. Scale bar = 3 cm

The alternance of darker and lighter very thin layers (0.2 mm) gives the schistic appearance. Microscopically, the rock displays a nematoblastic texture and consists mainly of amphiboles, chlorite and feldspar arranged in parallel bands (**Fig. 7**). Subordinately, quartz, opaque minerals and apatite may occur.

Amphiboles form rarely small xenomorphic grains, more often occur as needles or fibers up to 10–20 microns in length, with green-bluish to blue-yellowish or green pleochroism. The γ/c extinction angle is 22^0.

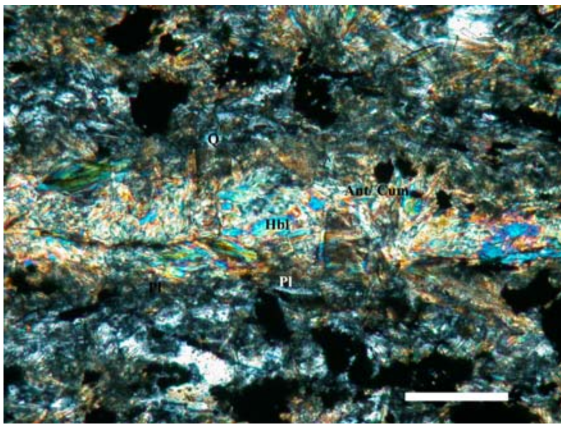

Fig. 7. Optical microphoto of the thin section through the amphibolite axe, showing amphiboles + chlorite bands (Hbl, Cl) alternating with quartz + plagioclase bands (Q + Pg). Other abbreviation: A/C – Anthophylite/Cummingtonite. Crossed nicols. Scale bar = 0.15 mm.

[4] A metamorphic rock, of greenish colour due to the high content of green amphibole (± chlorite). As amphibolite forms on the expenses of basic rocks (basalts), it may be called also metabasite.

Identification of the point analyses	Calculated structural formula	Mineral phase identified	Note	Related points on Fig. # 8
NV01E1	$(Ca_{1.69}Mg_{3.25}Fe^{2+}_{1.32}Al_{0.18}Fe^{3+}_{0.56})[Al_{0.74}Si_{7.26}O_{22}](OH)_2$	*Magnesiohornblende*	1	Hbl
NV01F1	$(Ca_{0.21}Mg_{3.37}Fe^{2+}_{3.05}Mn^{2+}_{0.09}Fe^{3+}_{0.31})[Al_{0.38}Si_{7.62}O_{22}](OH)_2$	*Cummingtonite/ Anthophyllite*	1	Ant/Cu m
NV01H1	$Ca_{0.5}Na_{0.5}[Al_{1.5}Si_{2.5}O_8]$	*Plagioclase*	2	Pl

Table 2. - Calculated structural formulae for minerals of the amphibolite axe. All phases were identified on group level by polarized light microscopy in the thin section prior to the standardless SEM+EDX analyses. Notes: 1 - calculated for 23 oxygens; 2 – calculated for 8 oxygens.

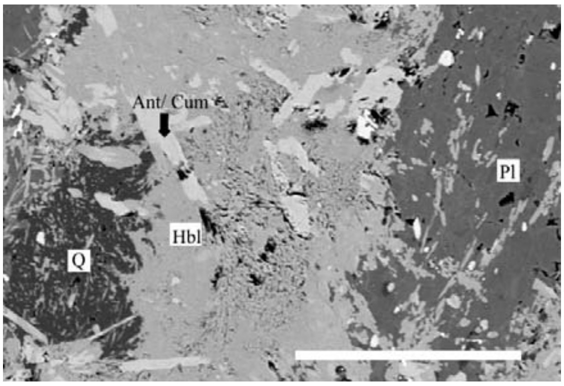

Fig. 8. - BSE image of the amphibolite axe (II.5). Abbreviations: Pl – Plagioclase; Hbl – Hornblende; Ant/Cum – Anthophylite/ Cummingtonite. Scale bar = 100 microns)

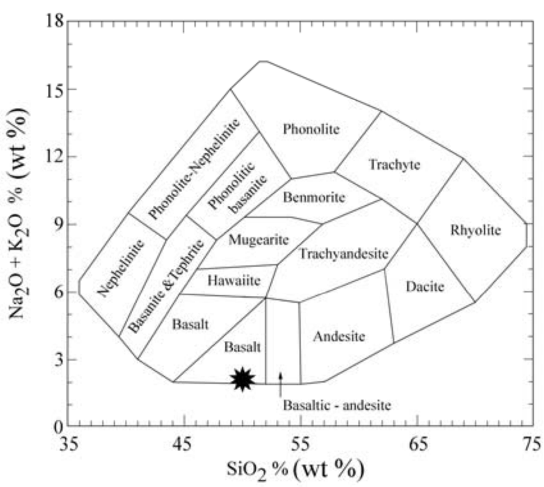

Fig. 9. - SiO_2 vs. Na_2O+K_2O discrimination diagram by COX *et al.* (1979). The black star represents the projection of Micula amphibolite chemistry into the field of basalts

The EMP analyses (**Table 2**) show the presence of both, magnesiohornblende and anthophyllite/cummingtonite[5] amphiboles (acc. to classification of LEAKE *et al.*, 1997). BSE image (**Fig. 8**) reveals that hornblende forms large crystals with inclusions of anthophyllite/cummingtonite. Hornblende is intergrown with plagioclase and sometimes with quartz. Feldspar (plagioclase) forms almost isometric grains (50–100 µm). The labradorite-andesine composition of plagioclase (**Table 2**) confirms the petrographic diagnosis for the rock as being amphibolite and not greenschist[6].

The chlorite lamellae show a pale green colour and have small size, between 5 and 10 microns. Among the opaque minerals, ilmenite prevails, compared with pyrite and chalcopyrite.

The PGA analyses of the amphibolite axe (**Table 3**) show a low SiO_2 content, around 50%, in the range of the basic rocks. Alkalies (Na_2O and K_2O) are also relatively low. In the SiO_2 vs. Na_2O+K_2O discrimination diagram by COX *et al.* (1979) the PGAA data on the Micula axe project in the field of basalts (**Fig. 9**). This composition shows that originally, amphibolite was a basic rock (basalts, dolerite, microgabbro).

The PGAA is rarely used for the lithic tools studies, excepting the works by RÉVAY *et al.* (2000), KASZTOVSZKY *et al.* (2002), SZAKMÁNY & KASZTOVSZKY (2004), KASZTOVSZKY *et al.* (2008), and so far no PGAA database for amphibolites is available for comparison. Our PGAA results for amphibolite axe were compared with reference data, e.g. archaeological findings together with rocks from outcrops, studied by SZAKMÁNY &

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[5] Due to the small size of the crystals, the distinction between anthophylite and cummingtonite was not possible.
[6] Amphibolites form at higher temperatures than the greenschists.

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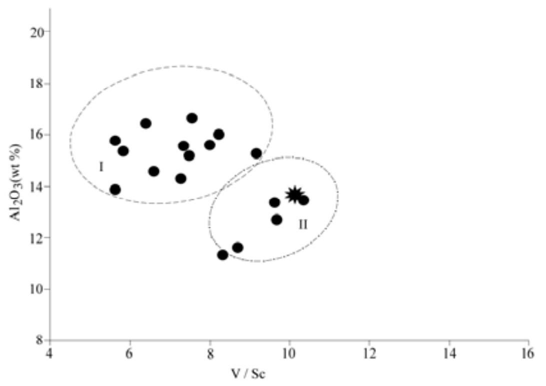

Fig. 10. - The projection of the Micula amphibolite (black star) PGAA data on the Al_2O_3 versus V/Sc diagram by SZAKMANY & KASZTOVSZKY (2004). The black circles represent the projection of SZAKMÁNY & KASZTOVSZKY (2004) data (Bohemian green schists), the black star is the projection of Micula data (amphibolite)

Table 3. - Prompt Gamma Activation analyses for the amphibolite axe. Major elements and relative error are given in %, trace elements in ppm. FeO_{TOT} as Fe_2O_3.

Element	Composition	Relative error	Detection limit
SiO_2	49.944	2.3	0.6
TiO_2	2.929	1.7	0.001
Al_2O_3	13.636	2.1	0.1
Fe_2O_3	13.359	2.1	0.06
MnO	0.211	2.1	0.007
MgO	6.515	5.3	0.2
CaO	9.663	2.9	0.04
Na_2O	2.022	2.2	0.02
K_2O	0.091	6.6	0.02
H_2O	1.293	1.3	0.007
Total	99.713		
B	0.2	1.4	0.00002
Sc	3.3	10.5	0.002
V	33.6	3.7	0.04
Cr	41.4	8.9	0.09
Sm	0.4	2.0	0.00005
Gd	0.6	3.6	0.000007
Dy	1.7	7.9	0.0005
S	81.5	8.2	0.06
Cl	3.4	8.1	0.001

KASZTOVSZKY (2004) and similar rocks and tools from the northern part of the Bohemian Massif (Železný Brod, Jizerske Hory Mts.; BRADÁK *et al.* 2005). According to Al_2O_3 versus V/Sc ratio, the authors (SZAKMÁNY & KASZTOVSZKY, 2004) inferred two groups of greenschists (**Fig. 10**):

group I, with high Al_2O_3 and low V/Sc ratio;

group II, with low Al_2O_3 and high V/Sc ratio;

Fig. 10 shows that the amphibolite axe found at Micula plots inside the group II, with its relatively low Al_2O_3 content and a high V/Sc ratio. According to the petrographic classification of SZAKMÁNY & KASZTOVSZKY (2004) the II.5 axe fits into the GS I and GS III groups which means a close relationship to the outcrop samples from the Northern part of the Bohemian Massif as well (BRADÁK *et al.* 2005).

Discussions: provenance remarks

From petrographic point of view, the Micula Neolithic axes are made from metamorphic rocks, very common in the area: hornfels and amphibolites. In order to find the possible sources for the raw materials, we compared our results with few chemical data available and further on, with the geological information.

The hornfels axe

This type of polished stone is relatively widely distributed in the archaeological material in whole Carpathian-Pannonian region (KALMÁR & STOICOVICIU 1990, HOVORKA *et al.* 2001; NIKL *et al.* 2002; SCHLÉDER & BIRÓ, 1999; SCHLÉDER *et al.*, 2002; SZAKMÁNY, 1996; STARNINI *et al.*, 2007). The hornfels tools represent in general only small part of all stone tools, but in the southeastern parts of the region they are relatively frequent (e.g. Endrőd and Szarvas sites in Hungary acc. to STARNINI & SZAKMÁNY, 1998 or Iclod site in Transylvania, Romania, acc. to KALMÁR & STOICOVICIU 1990). Hornfels are also widespread in the Vinča culture in Serbia (ANTONOVIĆ, 2006).

There are not many chemical analyses of the hornfels tools, but the mineralogical composition and appearance of the Micula tool is quite similar to those described from Neolithic Lengyel culture of Svodin (HOVORKA *et al.* 2001), or from the Neolithic locality in Ecsegfalva (STARNINI *et al.*, 2007).

Very similar in appearance, Neolithic polished stones were found at Suplacu de Barcău (Berettyószéplak) (**Fig. 11**), in the Northern Apuseni Mountains (LAZĂR *et al.*, 2007). The Suplacu de Barcău tools reveal a strong macroscopical and microscopical resemblance with the Micula ones, thus suggesting a possible similar provenance. The Suplacu de Barcău hornfels axe is greenish-dark grey in fresh cut and has also a weathering brownish cover as the Micula axe has.

Additionally, a slight schistosity is characteristic for both, the Micula and the Suplacu de Barcau tools.

Microscopically, the Suplacu de Barcău axe shows a similar aspect, with a fine-grained mass of pyroxenes, quartz and feldspars and rare pyrite crystals.

The geological sources for hornfels in the area are numerous (**Fig. 11**). Various hornfels occur in the north-western part of the Apuseni Mnts., mainly in the contact thermal aureolas of the Late Cretaceous-Early Paleogene magmatics (so-called banatites) and sediments such as siltic clays, clays (Geological Map of Romania; IONESCU & BALABAN, 1998). On the other hand, the hornfels could originate from the Rodnei Mts. as well where hornfels were described at the contact of clayish sediments and Neogene andesites (MOSONYI, 1998).

Hornfels formed also at the contact between Neogene volcanics and Pannonian siltic clays at Seini and Oligocene marls and clays at Firiza (Geol. Map of Romania, 1:200,000, Seini sheet).

In the alluvial sediments of all rivers crosscutting these areas, such as Someş, Vişeu, Criş, hornfels pebbles and boulders are common.

The amphibolite axe

Similar rocks in appearance, greenschists[7], were largely used for tools in the Carpathian–Pannonian region and in its surroundings (SZAKMÁNY & KASZTOVSZKY, 2001, 2004).

The comparison of Micula amphibolite with the greenschists described by SZAKMÁNY & KASZTOVSZKY (2004) shows that our sample plots in the second group (**Figs. 10**). This is due to the fact that these rocks, even very similar in appearance, represent two distinct petrographic types and the ratios between Al_2O_3 and V/Sc or alkalies vs. TiO_2 can be almost identical for different rock types and this is not relevant.

Nevertheless, east of the Micula site there are occurrences of both green schists and mainly amphibolites (MOSONYI, 1998; MURESAN, 2000). **Fig. 11** displays some of the occurrences of amphibolites in the NW Romania.

Amphibolites can be found in the northern part of the Eastern Carpathians, in the Rodna Mts. (MOSONYI, 1998, BALINTONI, 1997). Not far to the east, there are several occurrences of amphibolites in the Crystallino-Mesozoic zone of the Eastern Carpathians. The Eastern Carpathians in their northern part are crosscut by rivers such Someş or Tisa which might transport boulders of amphibolites.

In the northern part of the Apuseni Mts., in the Meseş and Plopiş Mts., as well as in the northern part of the Bihor Mts., amphibolites can be found (**Fig. 11**). The alluvia of the Crasna and Cris rivers contain frequently amphibolite boulders.

Together with the hornfels tools, at Suplacu de Barcau were found amphibolite axes (LAZĂR *et al.*, 2007). Similar with the hornfels axe, the amphibolite axe shows a strong macroscopical and microscopical resemblance with the Micula one, thus suggesting common sources. The Suplacu de Barcau amphibolite axe is dark grey in fresh cut and has an obvious schistuozity due to the fine white bands of feldspars.

Conclusions

Based on optical microscopy, X-ray diffraction, Electron Microprobe and Prompt Gamma Activation analyses, several data on the mineralogy, petrography and chemistry of the hornfels and amphibolite Neolithic axes found at Micula (NW Romania) resulted.

The comparison with the references data and the geological background including the petrography of the surrounding areas, points to a large range of possible sources: from the northern part of the Eastern Carpathians, to the northern part of the Apuseni Mts. However, the finding of almost identically Neolithic polished tools towards south, at Suplacu de Barcau, may indicate that the northern part of the Apuseni Mountains and the alluvial pebbles of Someş, Tisa, Crasna or Criş or rivers were most likely the ancient raw materials sources used for the tools.

On the other hand, other far-situated geological sources, such as the Bohemian massif, cannot be excluded, being known the long trade routes for tools in the Paleolithic and Neolithic (THORPE *et al*., 1984, CONSTANTINESCU *et al*., 2002, SIMON et al., 2003). The indication of possible sources is mainly theoretically. The further provenance investigations will focus on the study of samples from the neighbouring, such as hornfels and amphibolites in order to provide sound data for comparison.

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[7] Metamorphic rocks, very foliated, consisting of mainly chlorites.

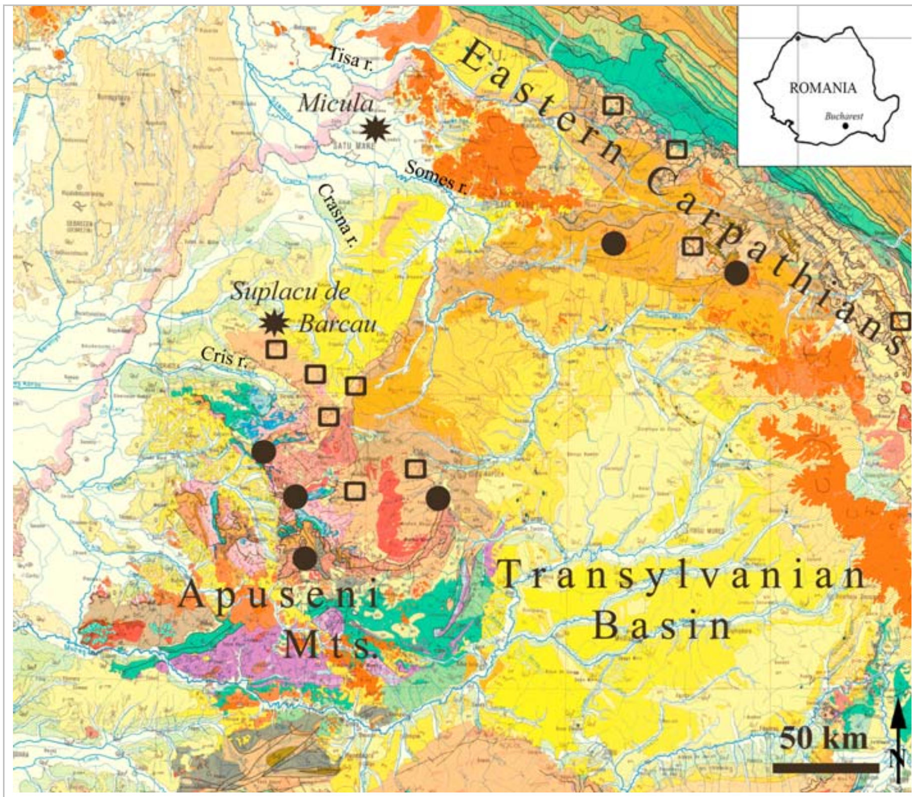

Fig. 11.

Possible geological sources for amphibolites and hornfels in North Romania (Transylvania). Legend: black stars – archaeological sites with amphibolite and hornfels axes; black dots – geological occurrences of hornfels; open squares – geological occurrences of amphibolites.

Based on the Geological Map of Romania 1:1,000,000 (1978) and 1:200,000 (Sheets: Satu Mare, Baia Mare, Vişeu, Oradea, Şimleu Silvaniei, Cluj, Bistriţa, Topliţa, Arad, Brad, Turda).

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*** GEOLOGICAL MAP OF ROMANIA, 1:1,000,000 and 1:200,000. Geological Institute of Romania. Bucharest.

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