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Palaeomagnetic and rock magnetic study of Lower Devonian sediments from Podolia, SW Ukraine: remagnetization problems

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SUMMARY

The Early Devonian segment of the Apparent Polar Wander Path (APWP) for Baltica was defined by seven poles almost all from British Islands. Among them only one was obtained for Devonian sediments from Ukraine. The lack of data from other parts of Baltica than Britain makes this segment of the path dubious. To improve this situation we carried out palaeomagnetic study of the Devonian sedimentary sequence in Podolia (Ukraine). The studied rocks represent grey limestone and red beds and were sampled in seven outcrops in the basin of the Dniester River. The standard palaeomagnetic experiments reveal Permian remagnetization corresponding to the pole position: 47°S/351.5°E for red beds and 45°S/340°E for grey limestone. The second component corresponding to the nearly Devonian pole position (2.3°S/338.4°E for red beds and 0°S/329°E for grey limestone) was determined as primary based on the fact that they were recognized in different sediments—limestone and sandstone and carried by different magnetic minerals: multidomain (MD) magnetite in grey limestone, and detritic grains composed of primary hematite with ilmenite intergrowths in red beds. The fabric of anisotropy of magnetic susceptibility of red sandstone and these samples of grey limestone which preserved Devonian magnetization are typical for sedimentary structure. The preservation of primary component of natural remanence was supported by analysis of mechanism of remagnetization and acquisition of secondary components. Hysteresis properties indicate that red beds underwent chemical remagnetization whereas grey limestone were not altered. Variscan orogeny caused the elevated heat and fluid flows along the edge of the Trans European Suture Zone and deep burial of Podolia. We suppose that red beds underwent chemical remagnetization caused by fluid flows, whereas grey limestone could acquire thermoviscous remanence due to elevated temperature caused by burial. Hot fluids could easily penetrate into sandstone and cause chemical alteration and creation of new iron oxides. As detrital hematite is chemically very stable it could preserve original magnetization. As grey limestone cemented at the beginning of digenesis was not penetrable by fluids during burial their remagnetization was caused mainly by increase of temperature and was not accompanied by alteration of rock. MD magnetite present in grey limestone could easily acquire thermoviscous magnetization. The remagnetization pattern is in agreement with the thermal history of Lower Palaeozoic rocks from Podolia. Maximum palaeotemperatures during burial reached 200 °C. The age of maximum palaeotemperature is about 300 Ma in excellent agreement with the position of poles calculated from remagnetization on APWP.

Key words: Magnetic fabrics and anisotropy; Palaeomagnetism applied to geologic processes; Remagnetization; Rock and mineral magnetism; Europe.

INTRODUCTION

Published by Torsvik *et al.* (2012) the Apparent Polar Wander Path (APWP) for Europe differs significantly from the previous one presented in the paper by Torsvik & Smethurst (1999). There are particularly great differences for the Late Silurian–Early Carboniferous time span. This segment of the APWP is very poorly defined, as the available palaeomagnetic data, although of good quality, are not

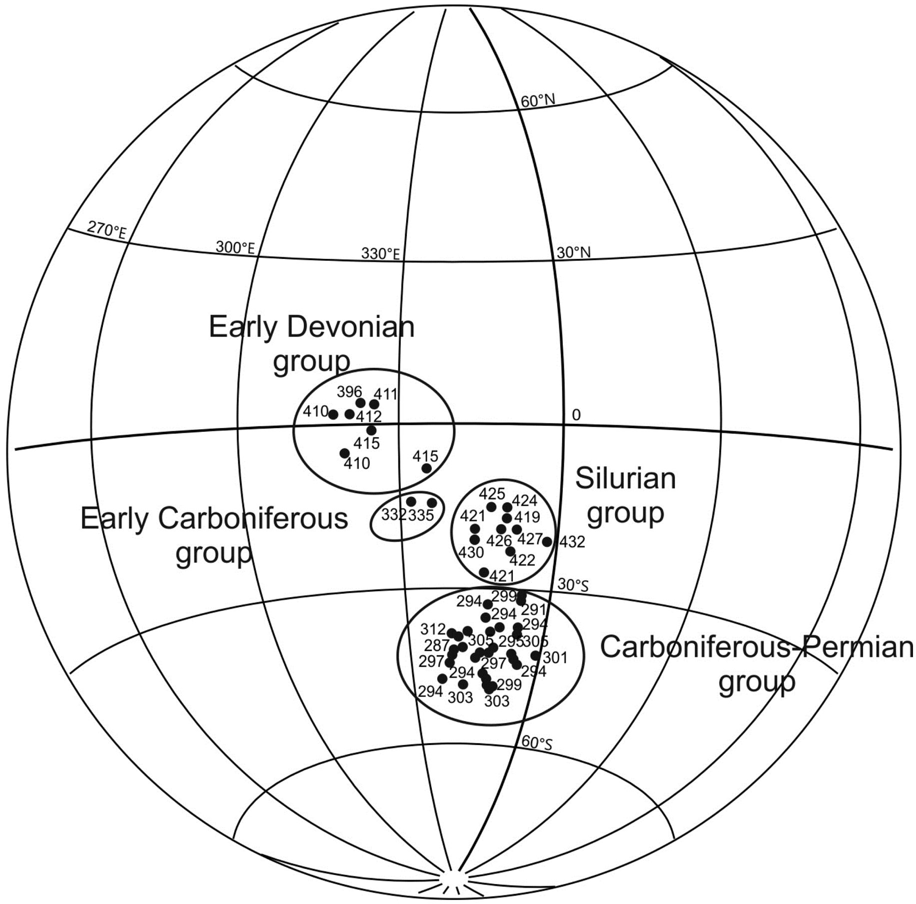

Figure 1. Carboniferous, Devonian and Silurian poles listed in Torsvik *et al.* (2012) as reliable data.

sufficient in number. A particularly poorly evaluated part of the curve is obtained for the Late Middle Devonian–Late Carboniferous time.

It is seen in Fig. 1 that the Silurian–Carboniferous data cited by Torsvik *et al.* (2012) for the European Plate demonstrate a big gap between Early Devonian times and the Carboniferous–Permian boundary with only two Early Carboniferous poles and seven Devonian poles almost all from British Islands. Four poles of age between 415 and 396 my come from Scotland, two come from England, and one was obtained for Devonian sediments from Podolia, Ukraine (Smethurst & Khramov 1992). The complete lack of data from other parts of the Baltica makes this segment of the path dubious. The Silurian segment of the path is better defined, as it is based on four results obtained from Scotland, four from the Scandinavian Peninsula, and two from Podolia, Ukraine, reported by Jeleńska *et al.* (2005). They lie close together, forming a 'cloud', which suggests a lack of tectonic movement between the cited areas after the time of sedimentation. The Devonian–Late Carboniferous APWP for Europe is so poorly defined because the data were derived mainly from sedimentary rocks which were remagnetized in Permian. The secondary widespread magnetization obscured or removed primary one.

So, the aim of our study is to add new data from the other geographic region than British Islands to the data base for the discussed segment of the path. We decided to study the Devonian sedimentary sequence in Podolia along the Dniester River (Ukraine). These sediments have long attracted the attention of geologists and palaeontologists because of the quality of exposure on the high and steep slopes of rivers and valleys, almost horizontal attitude, weak metamorphism and abundance and variety of fossils. As the Early Devonian–Early Carboniferous time is characterized by widespread Permian remagnetization of global range together with the discussion of possibility of preservation of primary component we considered mechanism of remagnetization. It is important to understand the remagnetization mechanism in order to isolate proper primary component.

GEOLOGICAL SETTING

Palaeozoic sediments are widespread in the west and southwest of the East European Platform. They are presented by marine and terrigenous rocks, the thickness of which increases moving westwards. Geologically, the Podolia region in southwest Ukraine represents the Dniester segment of the Peri-Tornquist margin of the East European Craton, and is bound to the west by the Trans European Suture Zone (TESZ). The western slope is characterized by shallow, dipping basement rocks. The fundament is characterized by gradual subsidence from east to west. In this direction the total thickness of sedimentary rocks is increasing. The rocks nearly form a monocline with a northwest strike.

Two structural stages separated by a significant gap in sedimentation and differences in structure are allocated. The first stage is composed of Upper Proterozoic (Vendian) and Palaeozoic (Lower Cambrian, Upper Ordovician, Silurian and Lower Devonian) rocks. These deposits are exposed in the valley of the Dniester River and its northern tributaries. Palaeozoic sediments overlie the Vendian with stratigraphic and angular (1–3°) unconformity. The Silurian sediments overlie the Ordovician, Cambrian and, in some places, the Vendian deposits. The part of the Lower Devonian strata is characterized by small faults and flexures, which are observed in the sediments of the Mitkovskaya and Chortkovskaya suites.

The Ordovician, Silurian and Lower Devonian strata compose the main part of the Lower Palaeozoic succession. The Upper Silurian–Lower Devonian sediments from Podolia form one of the most complete sections in the world. The most complete sections, in the middle of the valley of the Dniester River, are outcropping from the village Goraevka (48.57°N, 27.04°E) in the east to the village Nizniev (48.94°N, 25.09°E) in the west (Fig. 2). The Silurian–Devonian rocks exposed along the Dniester Valley are cut by the river across the strike as a bend restricted by the Zvanchik river valley, where Uppermost Silurian underlies Lower Devonian strata. The whole profile includes grey and red beds of the Tiverskaya and the lower part of the Dniestrovskaya series, which correspond to the Lochkovian stage of Standard Chronostratigraphy and have a duration of about 8.4 Ma (Gradstein *et al.* 2013, Figs 2 and 3).

The upper, second stage, belonging to the Cretaceous (Cenomanian stage) and Neogene (Tortonian and Sarmatian stages), lies horizontally and lower sediments cut across it.

RESEARCH OBJECTS

The Silurian-Lower Devonian sediments are exposed in the middle Dniester area in the territory of the southwest margin of the East European platform (Nikiforova & Predtechensky 1977; Tsegelnyuk *et al.* 1983). The results of the palaeomagnetic study of Silurian grey beds were reported earlier by Jeleńska *et al.* (2005).

The Lower Devonian deposits are exposed to the west in the basin of the Dniester River. Further on the western territory, they are known from drilling. The Middle Devonian deposits lie mainly in the Lvov Palaeozoic Trough, but they are not exposed on the surface.

The Podolian section of Lower Devonian consists of two series: Tiverskaya and Dniestrovskaya. Tiverskaya is the continuation of the Silurian sedimentation, and is represented by limestone and argillites. It is divided into the suites of Khudikovetskaya, Mitkovskaya, Chortkovskaya and Ivanevskaya. The Khudikovetskaya and Mitkovskaya suites are presented by interbedding argillites and flaggy limestone with a total thickness of approximately 190 m; the Chortkovskaya suite consists of argillites with interbedding organic-detrital limestone. The Ivanevskaya suite is characterized by a rhythmic alternation of argillites and clays, with layers of organic-detrital limestone, and a total thickness of up to 130 m. In this interval the single layers of red siltstone and argillites (from 0.5 to 3.0 m) appear everywhere.

The Dniestrovskaya series of red beds (approximately 440 m) was accumulated on the lowland plains in an arid climate. The sedimentation conditions are typical of a piedmont trough structure. The red beds are presented in the form of an alternation of mudstone, siltstone and sandstone. The Dniestrovskaya series consists of Ustechkovskaya, Khmelevskaya, Stripskaya and Smerklevskaya suites, which reach a thickness of about 1100 m to the west, in the Lvov Palaeozoic Trough. More detailed sedimentary logs and further geological information are reported by Tsegelnyuk *et al.* (1983).

For the palaeomagnetic study, seven outcrops were sampled (Figs 2 and 3). The Tiverskaya series is represented by five outcrops from the Khudikovetskaya and Ivanevskaya suites (III–VII) and the Dniestrovskaya series is represented by two outcrops from the Ustechkovskaya suite (1, 2). The total thickness of outcrops III–V is approximately 60 m, and the thickness of outcrops VI–VII is approximately 120 m. The total thickness of outcrops 1 and 2 is 50 m, and represent the whole Ustechkovskaya suite. The sampling rocks comprised grey limestone not studied before by Smethurst & Khramov (1992) and red sandstone collected in outcrops not previously studied.

A total of 173 oriented samples from seven localities were drilled or collected as hand samples for palaeomagnetic studies: 47 hand samples from the Tiverskaya series and 126 drilled and hand samples from the Dniestrovskaya series. A magnetic compass was used for the orientation of samples, as rocks were too weak for the need of a sun compass.

Standard palaeomagnetic experiments were performed in two laboratories—in the Institute of Geophysics, Polish Academy of Science, Warsaw and in the Institute of Geophysics of the National Academy of Sciences of Ukraine, Kiev. In both laboratories all measurements have been made inside magnetically shielded rooms within a magnetic cage made by Magnetic Measurements (UK). In Warsaw measurements were performed using 2G SQUID DC (USA) with a device for alternating field demagnetization attached to the SQUID. In Kiev, a JR-6 spin magnetometer with LDA-3A AF demagnetizer made by AGICO (Czech Republic) was used. In both laboratories, a non-magnetic furnace, MMTD80, made by Magnetic Measurements (UK) was used for thermal demagnetization. Possible mineralogical changes were monitored by measurements of magnetic susceptibility (*Km*) at room temperature after each step of thermal demagnetization. Anisotropy of magnetic susceptibility (AMS) was measured for all specimens with use of KLY2 and MFK1 kappabridges (AGICO).

Demagnetization results were analysed using the principal component analysis (Kirschvink 1980) by means of a PDA program package (Lewandowski *et al.* 1997) and Remasoft 3.0 software (Chadima & Hrouda 2006). Magnetic anisotropy parameters were calculated using the program Aniso (Jelinek 1977) according to Tarling & Hrouda's (1993) definition.

PALAEOMAGNETIC RESULTS

Tiverskaya series (grey sediments)

The Tiverskaya series consisted of grey limestone and comprised two suites: Ivanevskaya and Khudikovetskaya. Forty-seven hand samples were cut to 69 specimens, which were demagnetized either by AF or thermally. The preliminary results for grey sediments were described by Bakhmutov *et al.* (2012). The values of susceptibility were low, ranging from 40×10^{-6} SI to about 200×10^{-6} SI. Correspondingly, the values of NRM were also low, from 0.1 to 2.0 mA m^{-1}, making measuring procedures and interpretation of data difficult. Examples of demagnetization experiments by AF and temperature, together with changes of Km after consecutive heating steps, are presented in Fig. 4. The results show that Km usually increased after heating above 400 °C. Thermal cleaning was possible up to a temperature of 420–460 °C. At higher temperatures the intensity of NRM became too weak to be measured, or increased rapidly because of mineralogical changes. Sometimes, better results were obtained from AF demagnetization. During the demagnetization procedure, the majority of samples behaved as shown in Figs 4(a) and (b), where it is seen that a stable

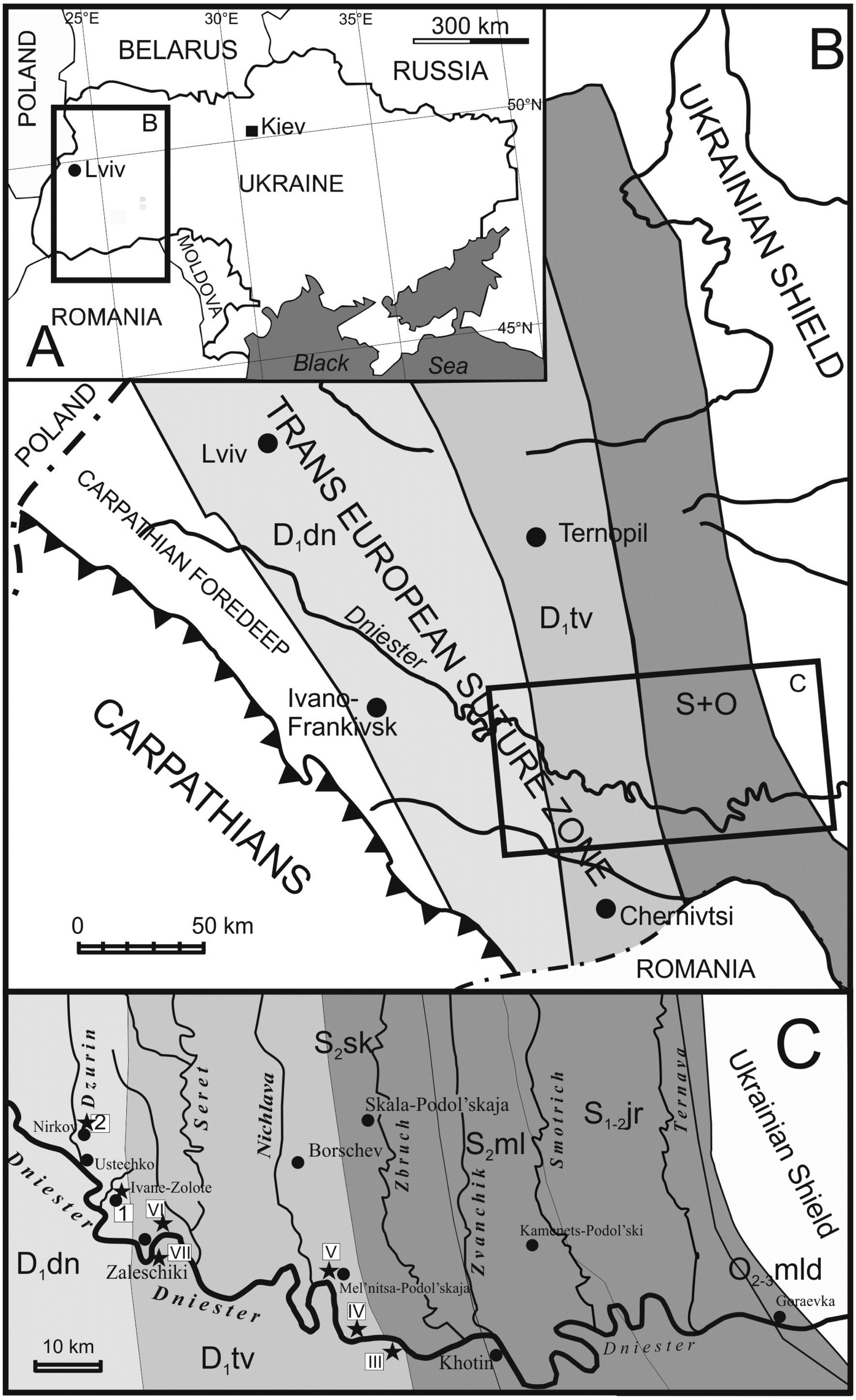

Figure 2. Geological sketch map of studied rocks and location of sampling area. The solid stars with numbers indicate the Devonian sections: grey sediments (Roman numerals) and red beds (Arabic numerals). The open stars with letters indicate sections of Silurian grey beds reported earlier by Jeleńska *et al.* (2005).

reversed component was revealed after the removal of low-stability present-day remagnetization. This reversed component, named TP, with an SSW declination and a negative inclination, was well isolated by AF and thermal treatment, and very well grouped. However, in some samples another component was evidently recognized at the end of the demagnetization path. This component was found in only 14 samples from two localities of the Ivanevskaya suite in the AF range of 30–70 mT and the temperature range of 300–460 °C (Figs 4c and d). This component, named TD, had an SW declination and a positive inclination.

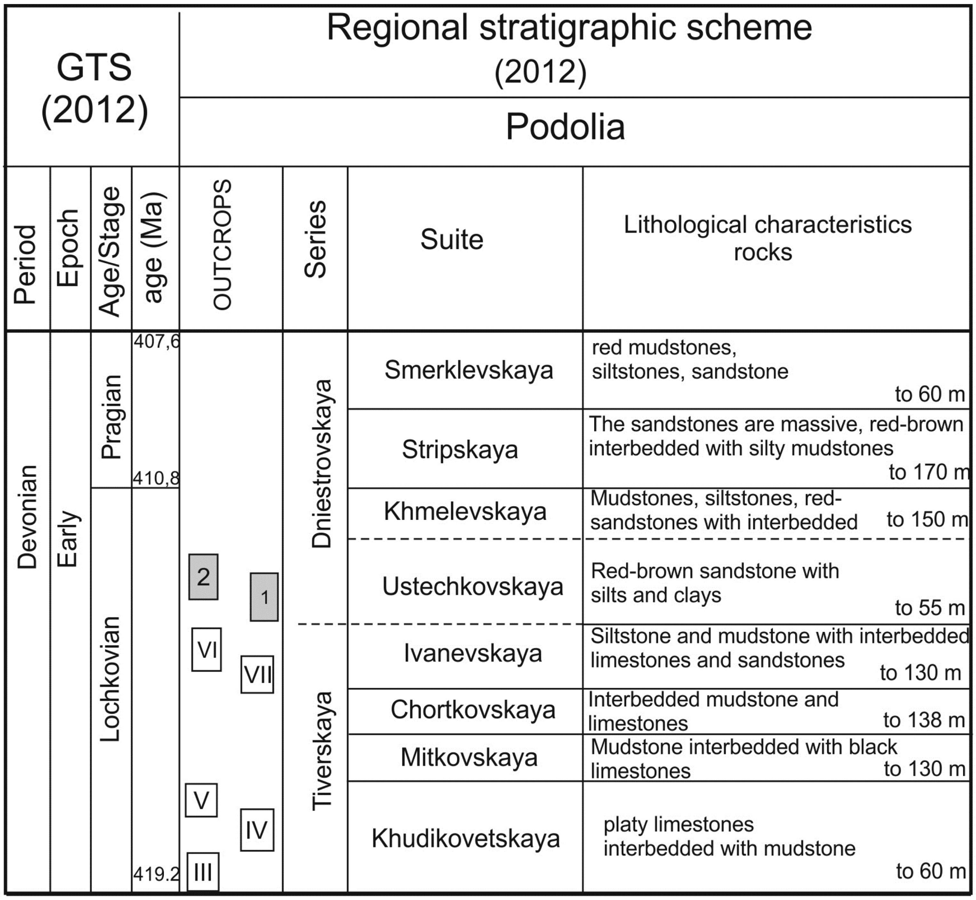

Figure 3. Stratigraphic scheme of Devonian rocks in Podolia compared with Standard Chronostratigraphy scheme (Gradstein *et al.* 2013). White (black) rectangles on the left-hand side show sections of grey (red) beds.

Dniestrovskaya series (red beds)

The Dniestrovskaya series consisted of red beds (sandstone) and comprised only one suite—Ustechkovskaya. Samples were taken from two localities along two profiles. The profile sampled in the outcrop in Nirkov represented the upper part of the Ustechkovskaya suite, and the profile sampled in the outcrop in Ivan-Zolote represented its lower part. Intensities of NRM and values of mean susceptibility were in the range of 1–10 mA m^{-1} and of 50 × 10^{-6} SI to approximately 200 × 10^{-6} SI, respectively.

The alternating field did not influence the NRM of the studied material (Fig. 4e). Therefore, all specimens demagnetized by AF were additionally demagnetized thermally. The results of thermal demagnetization obtained from both profiles usually revealed the presence of one or two components of NRM: a component of high stability (HT), named UP, removed in the range of 150/200–530/630 °C, and a component of very high stability (VHT), named UD, removed in the range of 590/610–690 °C (Fig. 4f). The intensity of the HT component was much higher than that of the VHT component. Susceptibility measured after the heating steps began to increase after heating to 500 °C; however, it did not involve any increase in NRM during the demagnetization procedure.

Standard computer packages often isolated only one component, with high intensity, instead of two, although the final segment of the demagnetization plot revealed the presence of the second, low-intensity component of different direction. So, we very thoroughly analysed those segments and found that the VHT components of NRM unblocked in the temperature range of 590–690 °C were present in an astonishingly large number of specimens. The intensity of these components was very low, but their directions were surprisingly well grouped. The first UP component had an SSW declination and an intermediate negative inclination, and was present in almost all specimens. The direction of this component was close to the direction of the TP component isolated in the grey limestone of the Tiverskaya series. The second VHT component had an SW declination and a positive inclination, and did not differ significantly from the TD component isolated in grey sediments.

Figs 5(a) and (b) presents the distribution of all isolated components from the Tiverskaya and Dniestrovskaya series, and Table 1 lists the isolated directions.

AMS measurements

AMS was measured for all specimens from the Tiverskaya and Dniestrovskaya series (Fig. 6).

Grey limestone from the Tiverskaya series (Fig. 6a) showed a low degree of anisotropy, *P*, not exceeding 1.05, and mixed directions of anisotropy axes. The maximum and minimum axes occupy horizontal as well as perpendicular positions. The same was observed for intermediate axes. This pattern was similar to the inverse anisotropy of SD magnetite grains. However, in our case we excluded inverse anisotropy because further rock-magnetic study

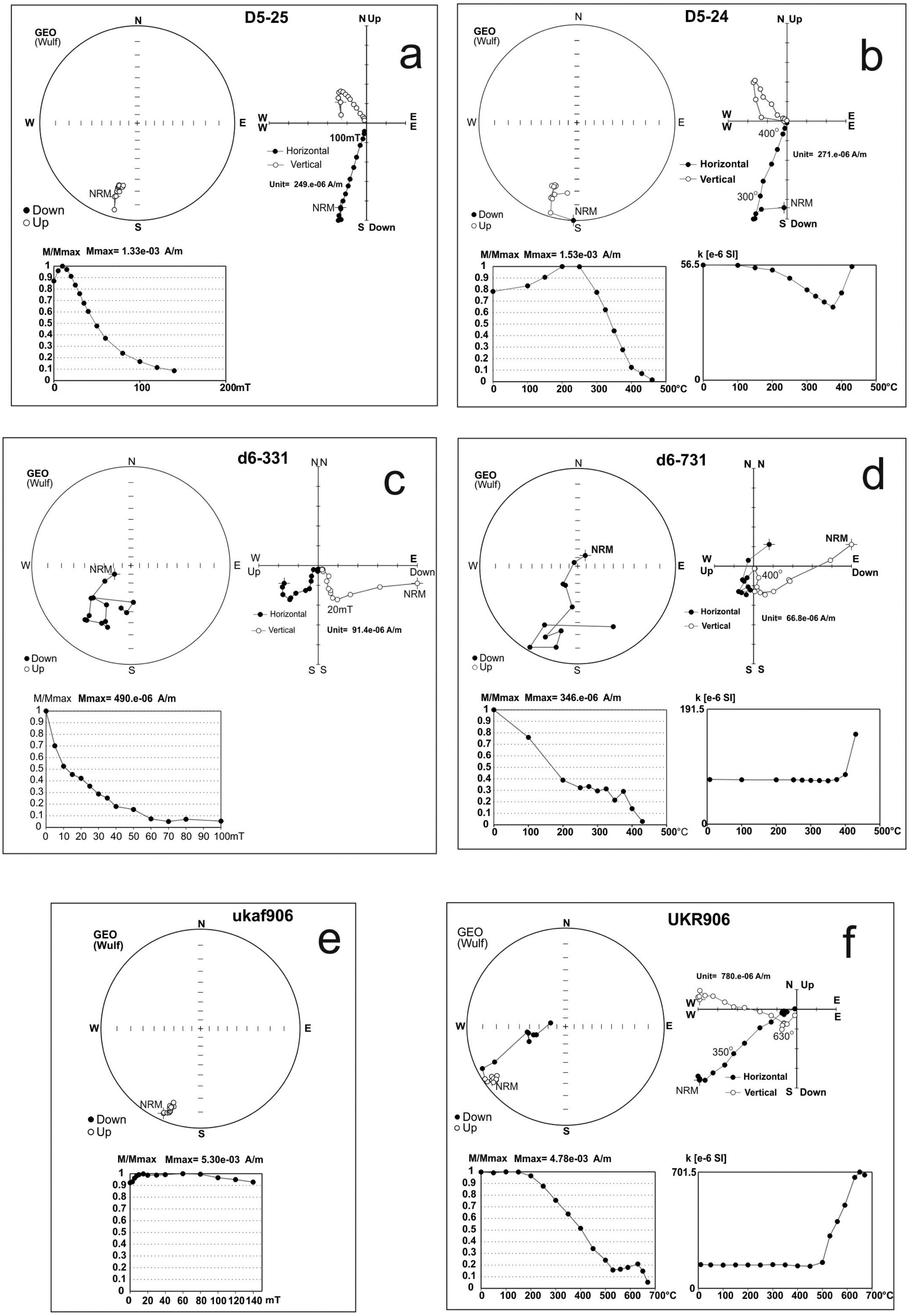

Figure 4. Stereographic projection of the NRM directions, orthogonal projections, decay of NRM normalized intensity during demagnetization and changes of magnetic susceptibility after each heating step for AF (a, c, e) and thermal (b, d, f) demagnetization of samples from grey limestone of Tiverskaya series (a, b, c, d) and red beds of Dniestrovskaya series (e, f).

revealed that the magnetite in the limestone of the Tiverskaya series was rather coarse-grained (multidomain, MD). The AMS axes for samples in which the Devonian component of NRM was isolated demonstrate sedimentary fabric with minimum axes perpendicular to the bedding plate and maximum axes lying in a horizontal position and distributed around a full circle (Fig. 6b). This suggests that sedimentary fabric was preserved in the samples in which the Devonian component was preserved.

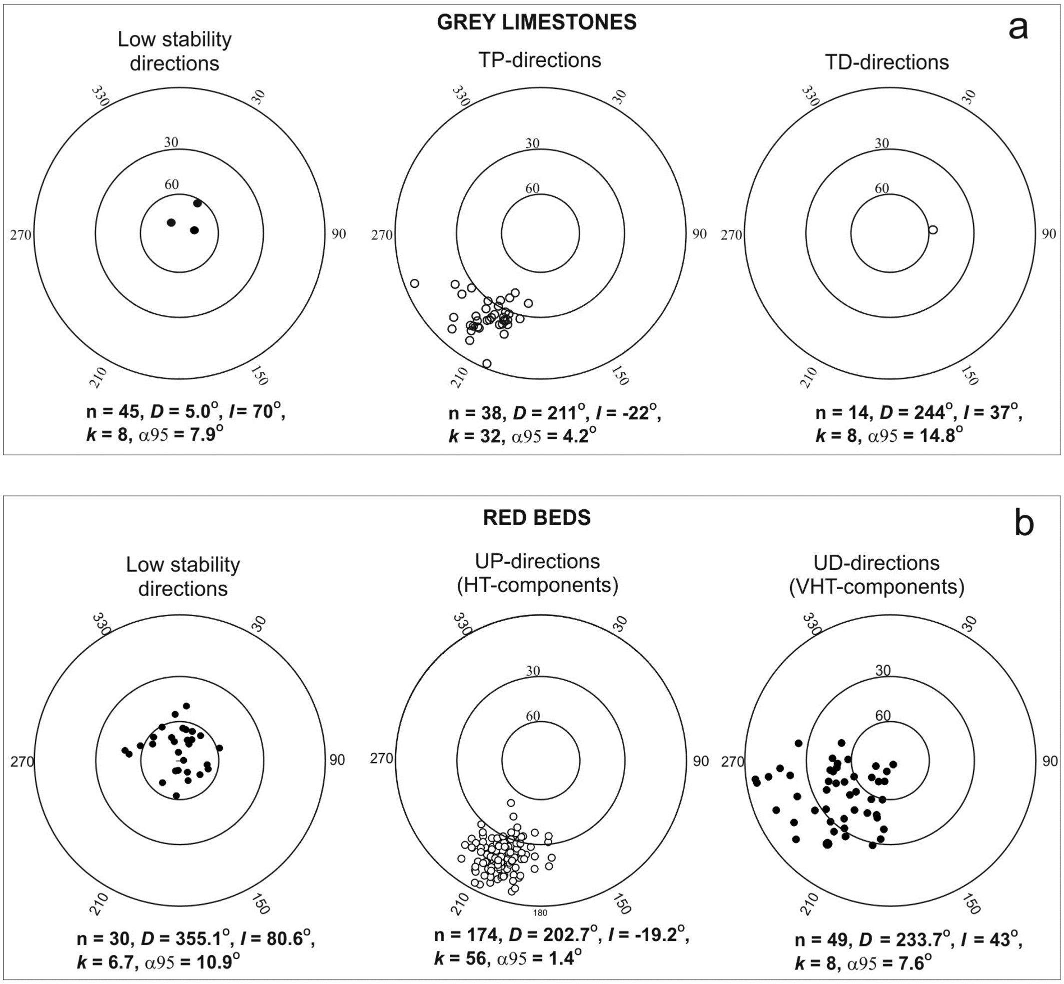

Figure 5. Stereographic projections of isolated palaeomagnetic directions for grey limestone of Tiverskaya series (a) and red beds of Dniestrovskaya series (b). Solid (open) circles indicate lower (upper) hemisphere projection.

Table 1. Mean palaeomagnetic directions and poles of the red beds from the Dniestrovskaya series (Ustechkovskaya suite) and grey limestone from the Tiverskaya series.

Components	Formations/series	*n*	*D*	*I*	α_{95}	*k*	P_{lat} (°N)	P_{long} (°E)
UD	Devonian red beds/ Dniestrovskaya	49	233.7	43.0	7.6	8	–2.3	338.4
TD	Devonian grey sediments/ Tiverskaya	14	244	37	14.8	8	0	329
UP	Devonian red beds/ Dniestrovskaya	174	202.7	–19.2	1.4	56	–47	351.5
TP	Devonian grey sediments/ Tiverskaya	38	211	–22	4.2	32	–45	340

Notes: n, number of samples used for suite mean calculation of NRM-components; *D* (°), *I* (°), α_{95} (°) and *k* are, respectively, the values of declination, inclination and statistical parameters associated with the mean value; P_{lat} (°N) and P_{long} (°E)—are latitude and longitude of the pole.

Red sediments from the Dniestrovskaya series revealed typical sedimentary AMS, with minimum axes perpendicular to both the bedding plate and horizontal maximum axes (Fig. 6c). The degree of anisotropy was low, with *P* not exceeding 1.048 and the positive shape parameter *T* indicating oblate fabric. The maximum anisotropy axes were grouped in an NNW–SSE direction, suggesting the influence of some ordering factor; for example, moderate current.

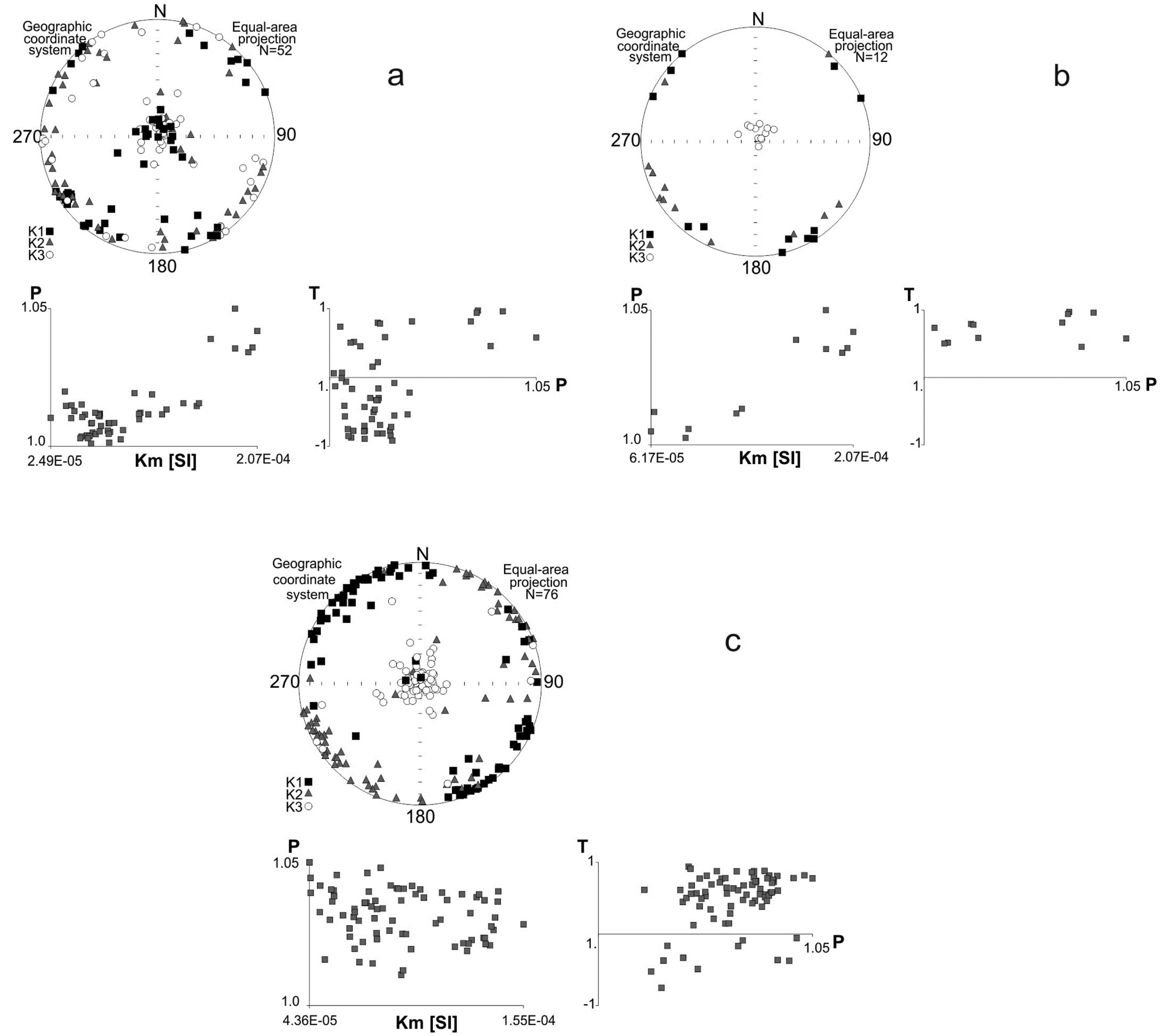

Figure 6. Stereographic projection of principal axes of magnetic susceptibility and magnetic anisotropy parameters: diagrams of *Km* versus *P* and *P* versus *T* for all grey sediments of Tiverskaya series (a), grey sediments of Tiverskaya series in which Devonian component is found (b), and red beds of Dniestrovskaya series (c). Squares—K1, triangles—K2 and circles—K3. $P = \mathrm{K1/K3}$; *Km* — magnetic susceptibility; *T*—shape parameter according to Tarling and Hrouda (1993).

ROCK-MAGNETIC INVESTIGATIONS

Methods

Rock-magnetic experiments performed in order to identify magnetic minerals—carriers of NRM—were made in the palaeomagnetic laboratory at the Institute of Geophysics in Warsaw. Identification of magnetic minerals and their structure was made by evaluating unblocking temperature (T_{ub}) and Curie temperature (T_c) values, hysteresis parameters, isothermal remanent magnetization (IRM) and anhysteretic remanent magnetization (ARM) acquisition curves, temperature dependence of susceptibility in the range of –196 °C to room temperature (low-temperature experiments) and in the range of room temperature to 700 °C (high-temperature experiments), and the Lowrie test (Lowrie 1990).

To determine T_{ub}, continuous thermal demagnetization of saturation isothermal remanence (SIRM) was carried out with the use of a device made by the TUS, Poland. SIRM was imparted to a sample in a field of 9 T and the remanence was measured during heating to 700 °C in a magnetic screen. The SIRM decay curves provided the spectra T_{ub} and allowed to identify magnetic minerals. The experiment was repeated twice in order to observe the changes of mineralogy imparted during the first heating.

Curie temperatures (T_c) of magnetic minerals were obtained by measuring changes of bulk susceptibility (Km) during continuous heating in air from room temperature (RT) to 700 °C and subsequent cooling to RT. This method revealed T_c of magnetic minerals present in heated specimens and those formed during heating. The experiments were performed with KLY-3S Kappabridge with a unit for

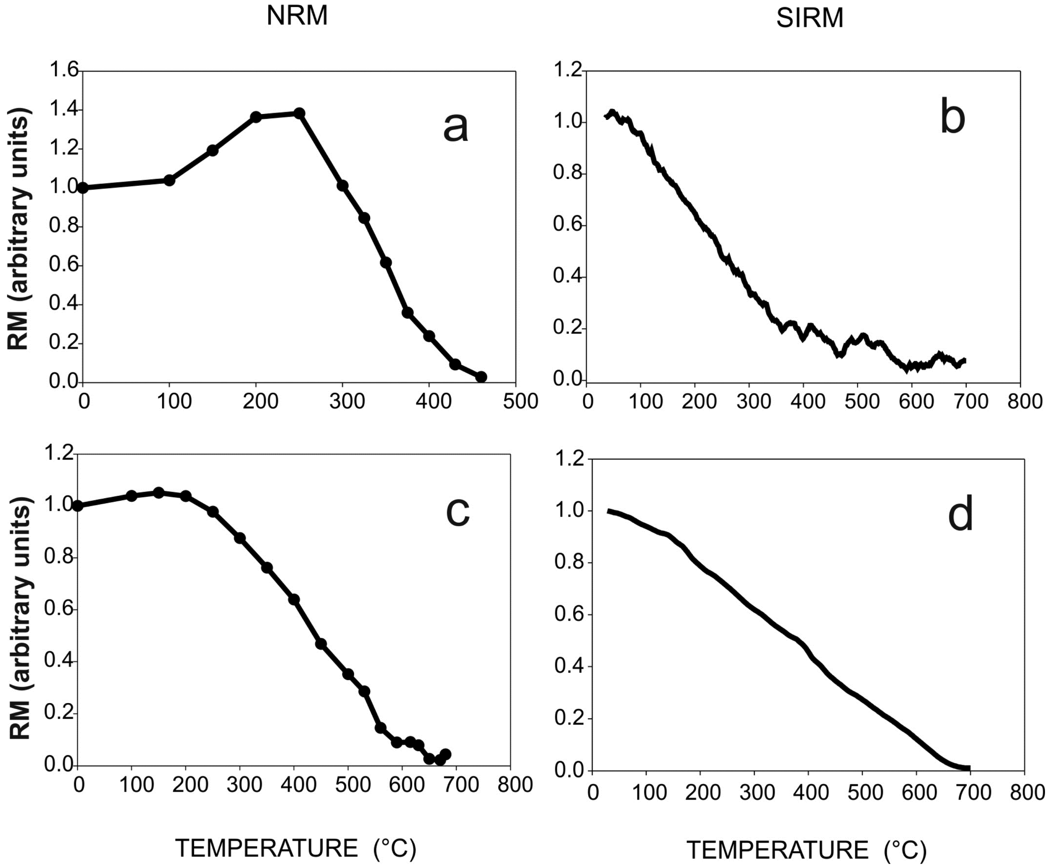

Figure 7. Examples of decay of NRM normalized intensity during thermal demagnetization and decay curves of saturation remanence (SIRM) during continuous heating for grey limestone (a, b) and red beds (c, d).

measurements of susceptibility in high (RT to 700 °C) temperature, made by AGICO, Czech Republic.

Low-temperature experiments, which provide information about the presence of magnetite and hematite if the Verwey (T_v) or Morin (T_m) transitions are observed, were not informative in our case.

The hardness and domain state of magnetic minerals were evaluated by measuring the hysteresis parameters: saturation remanent magnetization (SIRM), saturation magnetization (M_s) and coercivity (H_c). The coercivity of remanence (H_{cr}) was calculated from the back-field curve. Hysteresis loops were measured by means of a Vibrating Sample Magnetometer (Molspin, UK) or MicroMag (USA). Additionally, IRM and ARM acquisition curves were measured. Anhysteretic remanence was acquired using a Czech device, LDA-3, made by AGICO. A Lowrie test (Lowrie 1990) was carried out by magnetizing specimens in a steady field of 3.3 T in Z direction: 1.5 T in Y direction and 0.15 T in X direction.

Petrologic studies based on optical microscopy (Nikon), wavelength dispersive spectroscopy (Cameca SX 10) and scanning electron microscopy with EDS (JEOL JSM-6380LA) were performed in the laboratories in the Faculty of Geology at the University of Warsaw (Poland).

MAGNETIC PROPERTIES—IDENTIFICATION OF MAGNETIC MINERALOGY

Grey sediments have NRM directions carried by a medium coercivity mineral with an unblocking temperature characteristic of magnetite. SIRM (T) curves (Figs 7a and b) revealed a mineral with an unblocking temperature characteristic of magnetite; therefore, we accepted that magnetite was a carrier of both components of NRM.

Thermal demagnetization of red sediments revealed two components of NRM (Fig. 7c). The first component, UP, making up the main part of NRM, was present in almost all samples unblocked at a temperature of approximately 600 °C or lower. The second component, UD, was revealed in some samples at the end of demagnetization curves as a small tail unblocked at a temperature range of 590–690 °C. An unblocking temperature of approximately 690 °C pointed to hematite as a magnetic carrier. The other unblocking temperature (approximately 600 °C) of the first component was not as obvious for interpretation. The value was characteristic for non-stoichiometric magnetite; however, the extreme hardness of NRM, almost untouched during AF demagnetization, rather excluded both minerals: magnetite and maghemite. For a better understanding of the remagnetization mechanism, all rock-magnetic experiments aimed to identify the magnetic carrier of this component and its origin.

Thermomagnetic analysis of red beds giving a decay curve of saturation remanence SIRM during heating made for a whole rock gave T_{ub} characteristic of hematite. Other minerals were not observed; in particular, the mineral of $T_{ub} \approx 600$ °C was not seen (Fig. 7d). Heating to 700 °C did not change the composition of magnetic minerals present in the heated sample. Continuous heating of the strongest component of NRM made in the device for thermomagnetic analysis showed similar behaviour to its thermal demagnetization—the main part of NRM was unblocked at temperature $T_{ub} \approx 600$ °C.

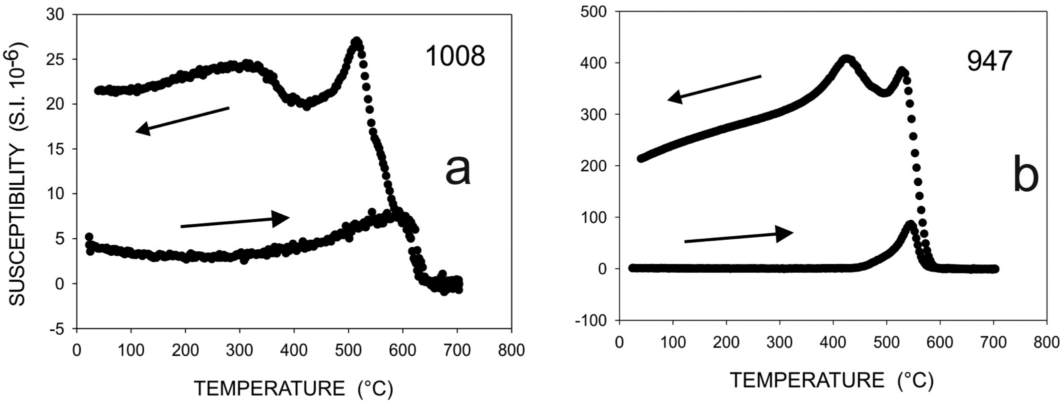

Figure 8. Changes of magnetic susceptibility (κ) during continuous heating (a, b) for red beds.

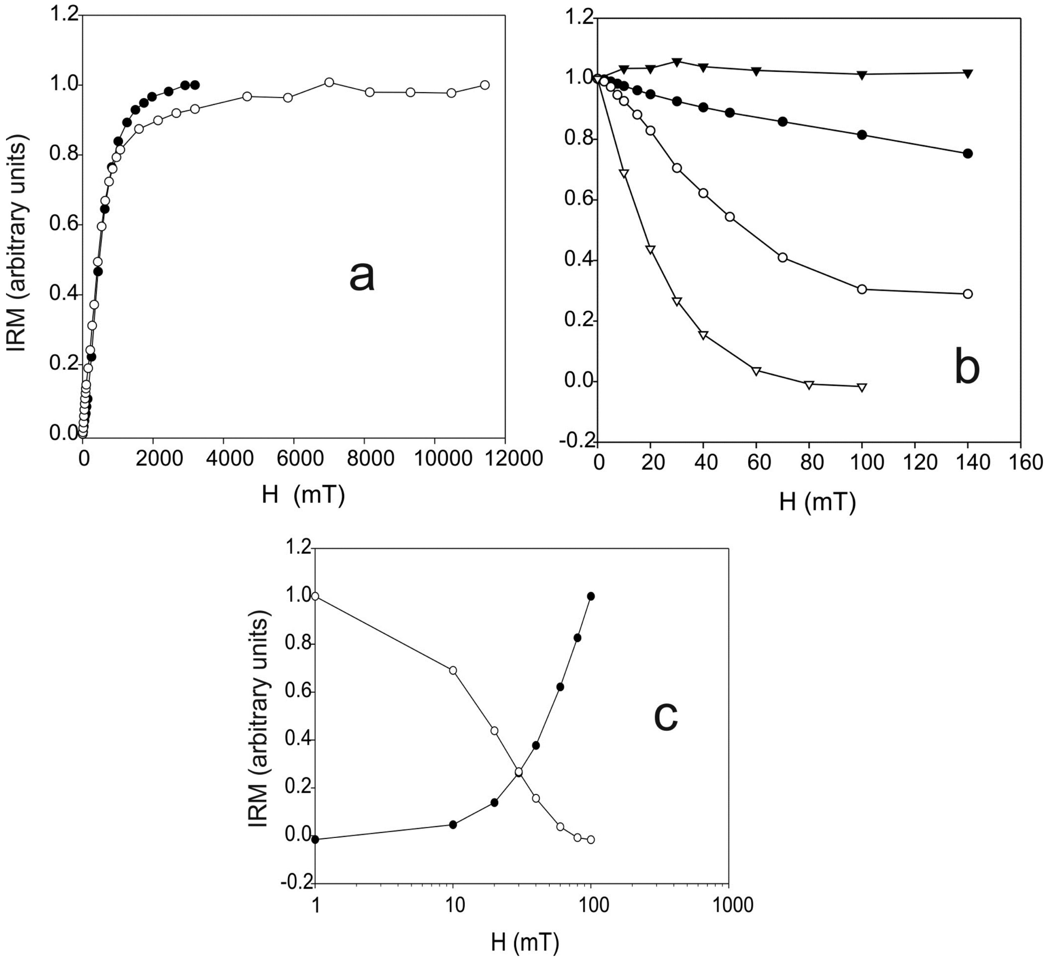

Figure 9. IRM acquisition curves (a), AF demagnetization of different remanence (b): NRM (full triangles); IRM acquired in 3.3 T (full circles); ARM acquired in 100 mT of AF field and 100 μT of DC field (open circles); IRM acquired in 0.1 T (open triangles), (c) example of IRM acquisition and demagnetization crossover plot for red beds. IRM is normalized to IRM value acquired at 100 mT field. The IRM crossing point lower than 0.5, theoretical value for SD magnetite and relatively high crossing field pointed to MD magnetite with presence of hematite (Symons & Cioppa 2000).

High-temperature behaviour of bulk susceptibility Km(T) performed for red beds was represented by two types of curves (Fig. 8).

(1) Km showed an increase at 600 °C and Curie temperature above this maximum of approximately 640–650 °C. During cooling, several magnetic phases were observed (Fig. 8a).

(2) Km showed an increase at approximately 530 °C and Curie temperature below 600–580 °C. During cooling, two peaks of Km were observed: a sharp peak of varying height at 530–500 °C and a second, broad one at a temperature of between 350 and 450 °C (Fig. 8b). Km(T) experiments showed reduction processes during heating, as hematite was not recognized. We supposed that hematite had been converted to magnetite.

IRM acquisition curves are characteristic for hard minerals. IRM did not reach saturation up to a field of 3.3 T (Fig. 9a). IRM acquired

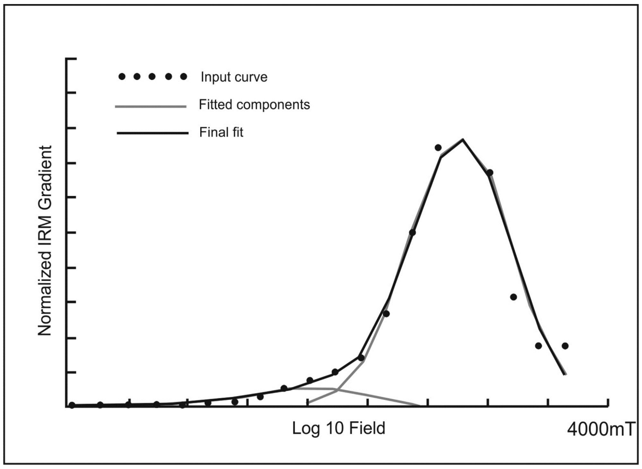

Figure 10. Example of cumulative log—Gaussian analysis of IRM acquisition curve for sample of red bed.

at the 3.3 T field was not demagnetized by AF up to 140 mT. Two specimens were magnetized to a field of 9 T. Even in such a high field, IRM was not completely saturated. This is in agreement with the high stability of NRM during AF demagnetization. Acquisition and AF demagnetization of IRM crossover plot (Symons & Cioppa 2000) performed for red beds shows MD magnetite behaviour revealing a small amount of soft magnetic mineral (Fig. 9c).

IRM acquisition curves were analysed using the cumulative log Gaussian method (Robertson & France 1994; Kruiver *et al.* 2001). The method discriminates minerals of different coercivity. For red beds samples, two or three components of remanence were isolated (Fig. 10). Table 2 gives detailed results of the analysis.

Hysteresis loops for red beds have a wasp-waist shape and great hardness, with H_c between 150 and 300 mT and H_{cr} between 300 and 500 mT (Table 3, Figs 11a and b). This is in contrast to the hysteresis loops for grey limestone, which show one component loop of soft coercivity (Figs 11c and d).

Ratios of M_{rs}/M_s and H_{cr}/H_c placed red sandstone in the area of the Day–Dunlop plot (Day *et al.* 1977; Dunlop 2002) occupied by a mixture of SD+SP belt (Fig. 11e). This region is distinct from the regions occupied by most other rocks and sediments and is usually associated with remagnetized carbonates (Jackson & Swanson-Hysell 2012). Hysteresis properties lead to the suggestion that in the studied red beds the dominant magnetic carriers behave similarly to SP and SSD grains characteristic of remagnetized carbonates. On the contrary, grey limestone lies in the region of MD grains with a small contribution of SD/PSD grains.

The Lowrie test (Lowrie 1990) performed for components acquired at field 3.3, 1.5 and 0.15 T showed that the main part of IRM was acquired at a field of 1.5 T. This is about 80–90 per cent of the total IRM value. A lower field of 0.15 T and a higher field of 3.3 T are not so effective in acquiring IRM (Fig. 12). The component of IRM acquired in the 1.5 T field showed the same unblocking temperatures as NRM: 530–580 °C and 650–680 °C, whereas two other components acquired in the 0.15 T field and in the 3.3 T field unblocked mainly at 660–680 °C.

Petrographic studies (Fig. 13) based on scanning electron microscopy (SEM), wavelength dispersive spectroscopy (WDS) and X-ray diffraction (XRD) analysis revealed that hematite was present in the red sediments in five forms: (1) detritic grains composed of primary hematite with ilmenite intergrowths ('tiger striped' grains, size from a few up to 100 μm); (2) martite grains (hematite pseudo-morphs after magnetite); (3) crystals of specular hematite within chlorite grains (up to 10 μm); (4) microcrystalline authigenic

Table 2. Fitted IRM component parameters. $SIRM_{R.C.}$ relative contribution of components to bulk IRM curve; $B_{1/2}$ field at which component acquires half of its saturation value; DP dispersion parameter; range $B_{1/2}$ ± DP.

Sample	Magnetizing field (T)	$IRM_{R.C.}$	$B_{1/2}$ log(mT) (mT)	DP log (mT)	Range (mT)
1008	3.2	0.078	1.673/47	0.400	19–118
		0.922	2.695/495	0.349	222–1107
966	3.2	0.277	2.569/371	0.745	67–2061
		0.723	2.704/506	0.287	261–979
1006	10	0.103	1.643/44	0.442	16–122
		0.897	2.848/705	0.467	240–2065
965	10	0.365	2.658/455	0.200	287–721
		0.635	2.664/461	0.795	74–2877

Table 3. Hysteresis data.

	Name	H_c (mT)	H_{cr} (mT)	M_s (μAm^2 kg^{-1})	M_{rs} (μAm^2 kg^{-1})	M_{rs}/M_s	H_{cr}/H_c
Red beds	1008	50.5	450.5	7446	2567	0.345	8.9
	1006	65.6	321.7	3029	1198	0.39	4.6
	966	61.1	263	4605	1599	0.347	4.9
	965	91.5	421	4072	1468	0.36	4.3
Grey limestone	VII 2/1	4.7	18	4871	274	0.056	3.8
	VI 3/5	3.5	12.3	19 830	899	0.045	3.5
	VI 7/2	7.3	22.1	4813	341	0.071	3
	V 2/4	9.6	39.5	2552	267	0.1	4.1
	IV 1	8.6	48.2	4055	445	0.11	5.6
	III 5	4.7	15.5	3122	158	0.051	3.3

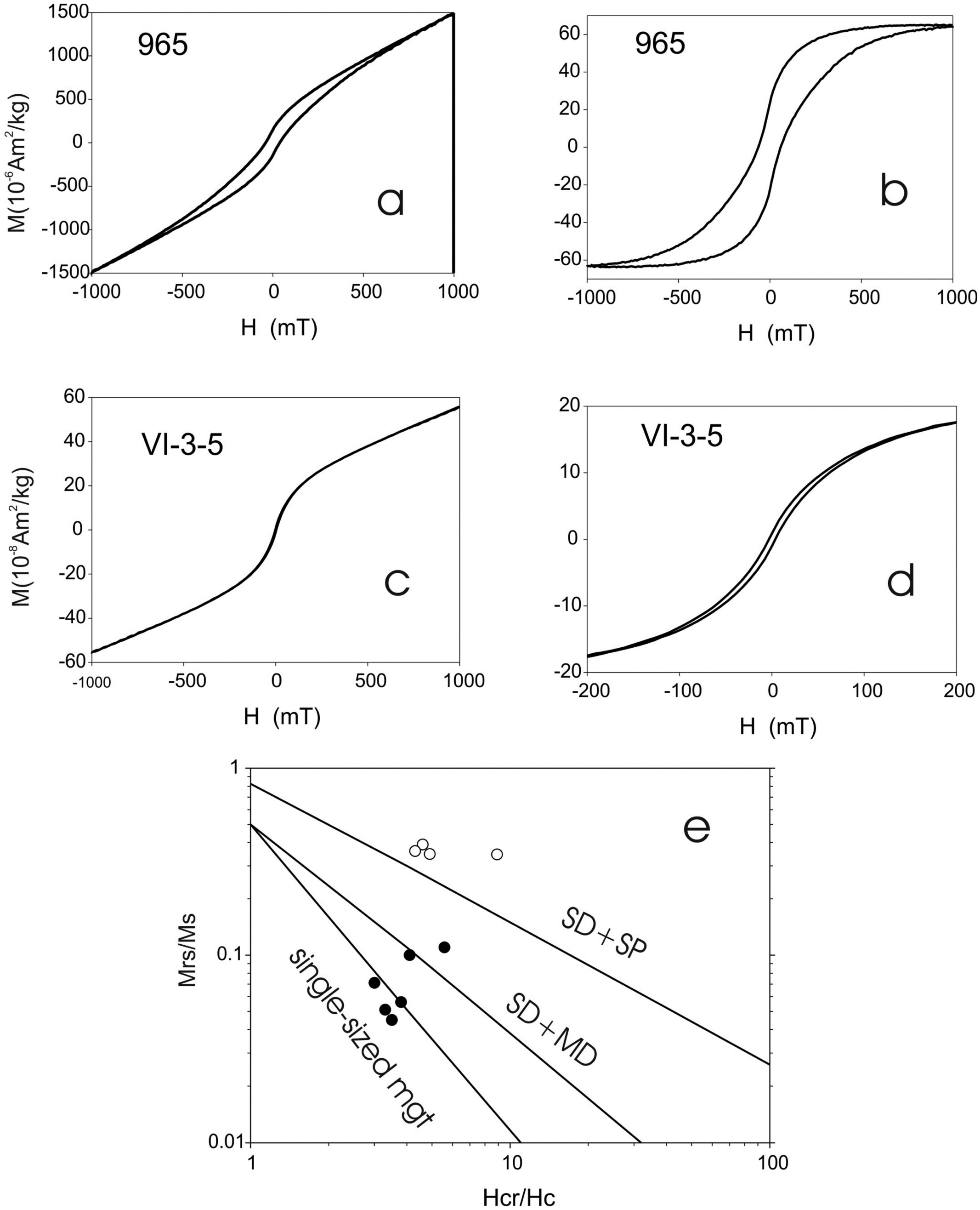

Figure 11. Examples of various hysteresis loops for red beds (a, b) and grey limestone (c, d). Wasp-waisted hysteresis plot for red beds is characteristic of remagnetized carbonates and related to the contribution of SP grains. Fig. (b) is corrected for paramagnetic contribution. Hysteresis plot for grey limestone is characteristic of MD magnetite. Fig. (d) shows enlarged middle part. (e) Day plot of that illustrates various configurations of grain mixtures (after McCabe & Chanell 1994) with hysteresis parameters for grey limestone (full circles) and red beds (open circles).

hematite (1–2 μm size) and (5) ultrafine pigment (cauliflower-like grains ∼100 nm size).

DISCUSSION

Both studied rock units revealed two components of NRM. The first component (TP and UP), recognized in almost all samples from the Ivanevskaya and Khudikovetskaya suites (Tiverskaya series) and from the Ustechkovskaya suite (Dniestrovskaya series), had an SSW declination and a negative inclination. Pole positions calculated from these directions lie in the Permian segment of APWP (Fig. 14) published by Torsvik *et al.* (2012) for Baltica/Stable Europe. We accepted these directions as Permian remagnetization. The second component (TD and UD) was isolated at the end of the thermal demagnetization path for red sandstone (Ustechkovskaya suite) and in only a few samples of grey limestone from the Ivanevskaya suite. This component had an SW declination and a positive inclination. Pole positions calculated from these directions lie close to the Devonian segments of APWP and to the Devonian pole position obtained by Smethurst & Khramov (1992) for Podole sediment (3.7°N, 325.5°E). In grey limestone, both components are carried

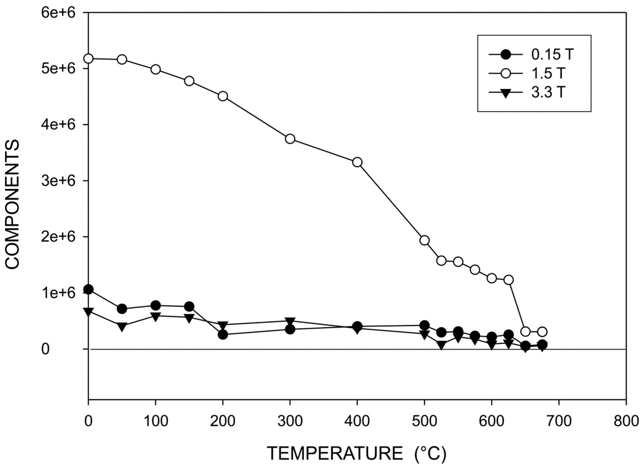

Figure 12. Example of thermal decay curves of tri-axial IRM imparted on red beds sample.

by magnetite grains. Among five forms of hematite revealed in the red beds by petrographic analysis, the only hematite of unquestionable detrital origin is the hematite with the ilmenite–hematite intergrowths ('tiger striped' grains, Fig. 13b), which is a product of high-temperature exsolution processes (Walker *et al.* 1981). Such grains must have been eroded from the crystalline basement of the Ukrainian Shield. Martite grains show clear evidence of oxidation of pre-existing magnetite. These grains contain ilmenite laths resulting from oxidation of the original titanomagnetite grains (Fig. 13d). However, petrographic analysis does not provide information about the time of the martitization process. Martite could have been deposited or created by post-depositional oxidation of the detrital magnetite grain (Van Houten 1968). Specular hematite in cleavage planes of the host chlorite grains is also a product of oxidation (Fig. 13c). WDS analysis shows that chlorite has formed as an alteration product of biotite. Oxidation of Fe-bearing silicates often yields specular hematite and textural evidence indicates sequences of reactions that are the result of *in situ* oxidation (Walker *et al.* 1981). The microcrystalline hematite (Fig. 13e) and ultrafine pigment (Fig. 13f) are unequivocally authigenic. They form cements between quartz, feldspar, muscovite and chlorite grains (Fig. 13a). Each of the above forms of hematite is coarser grained than the superparamagnetic threshold for hematite ($\sim$0.037 µm); therefore, each can contribute to remanent magnetization of the rocks. An unblocking temperature of 660–680 °C is usually attributed to specular or detrital hematite grains, which are the best candidates for being carriers of the Devonian component of NRM. An unblocking temperature spectrum of 580–650 °C points to fine-grained pigmentary hematite which can be the carrier of secondary remanence.

We shall discuss two aspects of the obtained results.

Possibility of primary directions

There are several facts supporting the hypothesis that TD and UD components are primary magnetization that recorded the Devonian palaeofield. First of all, they were recognized in different sediments—limestone and sandstone. They were carried by different magnetic minerals—magnetite in the case of the Ivanevskaya suite, and hematite in the case of the Ustechkovskaya suite. The origin of the natural remanent magnetization (NRM) in red beds remains a subject of debate. Some argue that hematite can carry a depositional remanent magnetization (DRM) and therefore a primary NRM (e.g. Tauxe *et al.* 1980; Steiner 1983), while others suggest that a chemical remanent magnetization (CRM) acquired long after deposition is the dominant mechanism, giving rise to a secondary NRM (e.g. Roy & Park 1972). Butler (1992) pointed out the 'red beds controversy': whether specular hematite is depositional or secondary and what kind of NRM it carries. Butler stated that in some red beds the specular hematite carries a DRM. In addition, Kodama (2012) presents the idea that the remanence in red beds can be depositional or early diagenetic. Besides specular hematite detrital grains of hematite with ilmenite lamellae are recognized by an electron microprobe in BSE (Back Scattering Electron) images. Such primary structure is a product of high-temperature exsolution processes. Magnetization of these hematite grains can originate from lamellar magnetism, which, according to Robinson *et al.* (2002, 2004) is characterized by extreme hardness and AF stability. Hematite from the Ustechkovskaya suite has extreme hardness. This suggests that detrital hematite carries primary DRM. Specular hematite can be a product of early diagenesis and can carry remanence not substantially different from the Devonian one. This conclusion is supported by the AMS fabric of red sandstone, which is typical for a sedimentary structure with minimum axes of AMS perpendicular to the bedding plate. Despite the sedimentary fabric of AMS we do not observed shallowing of inclination in red beds. The correction for inclination proposed by Torsvik *et al.* (2012) did not improve the Devonian pole position of red beds. Kodama (2012) gave several examples where inclination shallowing was not observed. Lack of inclination shallowing was reported by Sun *et al.* (2006); Schmidt *et al.* (2009) and others, and was attributed to coarse-grained hematite in the SD range, which has the highest Irs values (Dekkers & Linssen 1989; Kodama 2012).

Remagnetization

As our knowledge about remagnetization phenomena increased, we learned to use the accumulated data of remagnetized directions and properties of remagnetized rocks for a better understanding of remagnetization processes in order to isolate primary components, date diagenetic events and relate them to the thermal and geodynamic history of a region (Elmore *et al.* 2012; Jackson & Swanson-Hysell 2012; Van der Voo & Torsvik 2012). In our case we dealt with different rocks—limestone and red beds—remagnetized in approximately the same Permian time. The close geographic position of the studied rocks implied the same source of alteration. However, despite similar remagnetized directions obtained for both rock units, the mechanisms involved for acquiring secondary components were different. In grey limestone, usually only one component was isolated in a single sample. Permian remagnetization was recognized in samples in which Devonian direction was not preserved. The majority of magnetite grains was remagnetized. The remagnetization of grey limestone is not homogenous. There are a few spots where magnetite grains with primary sedimentary fabric and primary Devonian direction were preserved. It was not possible to recognize whether we have one or two generations of magnetite. The narrow hysteresis loops and field occupied by samples of grey limestone in the Day–Dunlop plot were characteristic of unaltered rocks. According to McCabe & Chanell (1994), they represent SD+MD grains or single-sized magnetite close to the MD line. A total of AMS fabric of grey limestone had a distinct distribution of anisotropy axis. They all lied in a horizontal, bedding plane or perpendicular to this plane. The degree of anisotropy was low. It looks like the AMS axes lie parallel to the directions of easy magnetization closest to the magnetic field. On this basis, we suppose the thermoviscous

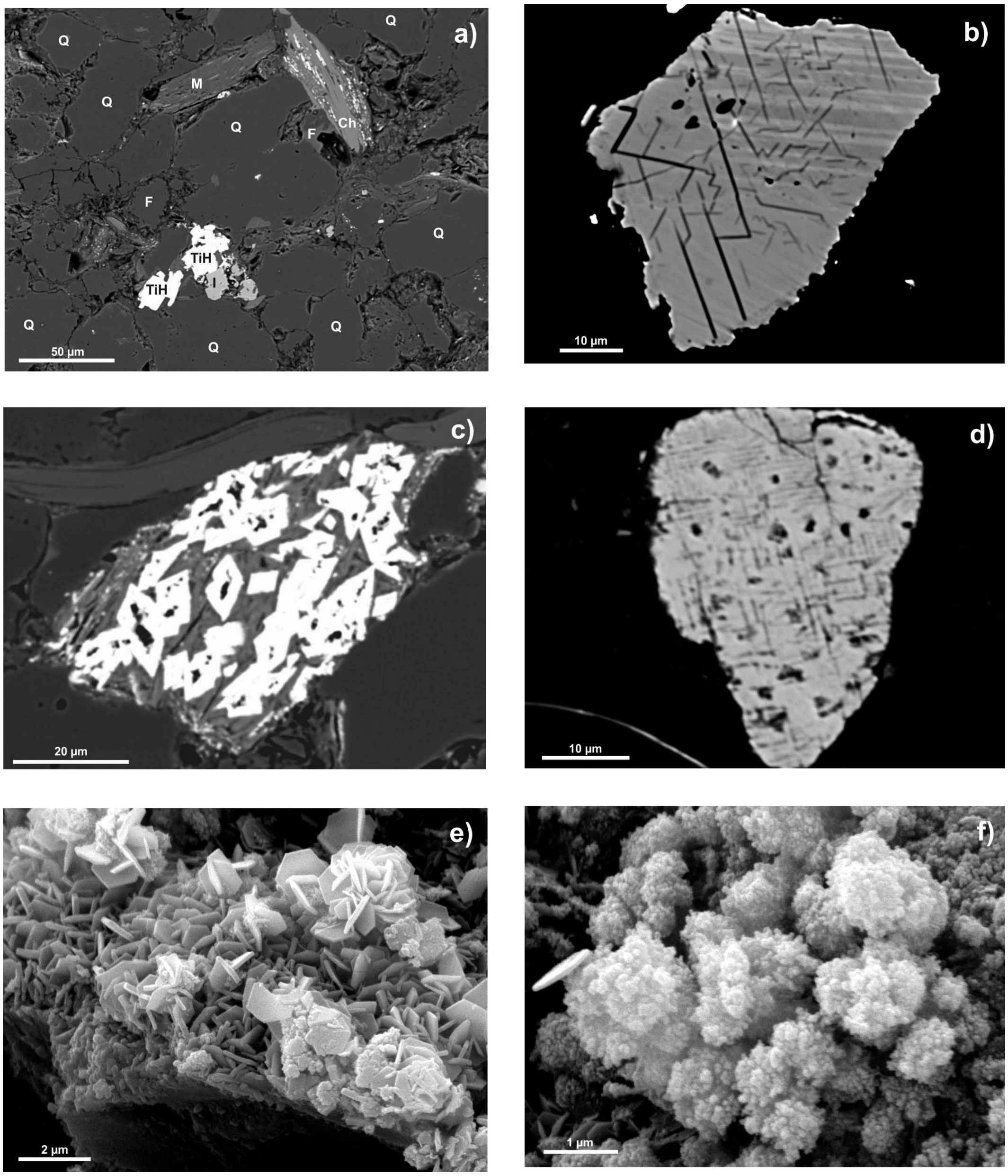

Figure 13. Magnetic carriers in the Devonian red beds: (a) BSE image of the red bed sandstone. Q—quartz, F—feldspar, M—muscovite, Ch—chlorite, TiH—Ti hematite, I—ilmenite; (b) BSE image of the detrital grain of primary hematite with ilmenite intergrowths ('tiger striped' grain); (c) BSE image of crystals of specular hematite within a post-biotite chlorite grain; (d) BSE image of martite grain; (e) SEM image of hexagonal autigenic hematite and (f) SEM image of cauliflower-like grains of hematite.

mechanism of remagnetization without significant alteration of the rock in grey limestone.

In red beds, Devonian directions were isolated as a second component at the end of the thermal demagnetization path. Authigenic, pure hematite crystals (1–2 µm in size) occurring in the ferruginous cement of sandstone were the main source of SIRM and were responsible for SIRM(T) behaviour which showed only the highest unblocking temperature of hematite at approximately 680 °C. But NRM thermal demagnetization revealed an unblocking temperature spectrum of 530–630 °C and the temperature near to the highest unblocking temperature for hematite. Lowrie experiments also demonstrated two T_{ub} values. IRM acquisition curves analysed by the cumulative log Gaussian method (Robertson & France 1994; Kruiver *et al.* 2001) identified two or three components of remanence associated with minerals of different coercivity in red beds. Although the T_{ub} of the second component was close to the T_{ub} of magnetite (≈600 °C), very high coercivity excludes magnetite or maghemite. The best candidate was secondary fine-grained pigmentary hematite. In addition to several generations of hematite in red beds, small amounts of soft magnetic mineral were detected by analysis of IRM acquisition curves using the cumulative log Gaussian method and by crossover plots of IRM acquisition and AF demagnetization curves (Symons & Cioppa 2000). Unidentified iron oxides recognized in altered chlorite grains can be this phase. They can record recent low-stability components of NRM. This phase is of low importance for palaeomagnetic results; however, it complies with the description of alteration processes in red beds.

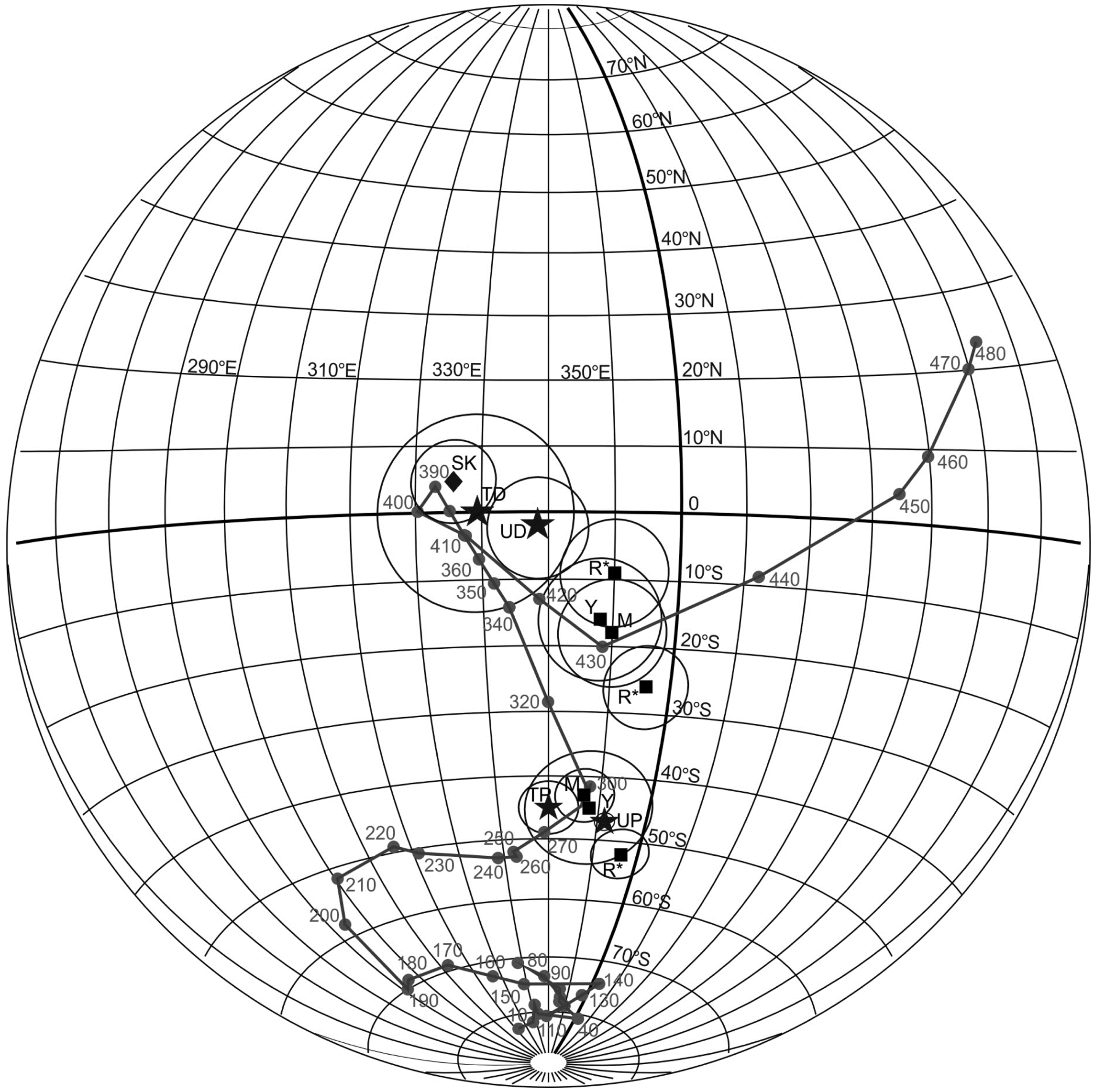

Figure 14. Reference APWP for Baltica on equal area projection (after Torsvik *et al.* 2012). Stars—Devonian (TD and UD) and Permian (TP and UP) pole positions from this paper; squares—Silurian and Permian (R*, M and Y) data for Podolian sediments (Jelenska *et al.* 2005); SK—Devonian pole from Smethurst & Khramov (1992).

The region in the Day/Dunlop plot occupied by hysteresis parameters for red sandstone (Day *et al.* 1977; Dunlop 2002) lies along the belt of a mixture of SD+SP grains and is usually associated with remagnetized carbonates (Jackson & Swanson-Hysell 2012).

The magnetization process for red beds samples can be summarized as follows. Initially, hematite and magnetite grains of detrital origin were deposited during the Early Devonian period. These two minerals carried the primary detrital remanent magnetization (DRM). After deposition the process of martitization (hematitization of magnetite) began, as well as precipitation of ferruginous cements between detrital grains. The newly formed magnetic minerals can carry the secondary 'early' CRM with the same directions as primary DRM. The second event of oxidation and CRM acquisition was caused by burial. The process was regulated by the availability of oxidizing fluids and the availability of Fe^{3+} for the continuing precipitation of secondary fine-grained haematite. During the Permian period, the region probably went through a post-Variscan fluid event that accelerated the formation of fine-grained hematite and ultrafine pigment. In addition, the process of chloritization of biotite began during this time. These newly formed magnetic minerals, particularly the specular hematite within the chlorite grains, carried the secondary 'late' CRM.

The remagnetization pattern is in agreement with the thermal history of Lower Palaeozoic rocks from Podolia described by Środoń *et al.* (2013). On the basis of XRD, K-Ar dating, apatite fission track (AFT) and percentage smectite (%S) methods, the authors concluded that Silurian and Lower Devonian rocks from Podolia underwent deep burial diagenesis with maximum temperatures reaching 200 °C. The advanced diagenesis of the Devonian–Carboniferous Dniester slope were related to Variscan orogeny and induced by Upper Devonian-Carboniferous overburden and a tectono-thermal event induced by the elevated heat and fluids flow along the edge of the TESZ. The sequence of sediments along the Dniester—the younger red beds lying near to the TESZ zone and the grey limestone lying just over Silurian rocks and buried deeper than younger units—justifies the assumption that the remagnetization of red beds could be caused by fluid flow, whereas grey limestone could be remagnetized due to elevated temperature caused by burial. Sandstone are pervious for hot fluids. In elevated temperature such fluids caused alteration of biotite and creation of chlorite with new iron oxides. This can explain chemical remagnetization of red beds. Grey limestone were deposited in marine environment and cemented soon after deposition and were prevented for fluid action. During burial remagnetization process was caused mainly by increase of temperature. This explains acquisition of thermo-viscous remanence. As relaxation time (Pullaiah *et al.* 1975) is strongly dependent on temperature or volume, remanence was blocked in narrow range of temperatute/volume during short time span. Low scatter of Permian directions support this conclusion. The age of maximum palaeotemperatures induced from K–Ar dating (about 300 Ma) by Środoń *et al.* (2013) is in excellent agreement with the pole positions (TP and UP) calculated from the secondary components (Fig. 14).

CONCLUSIONS

The standard palaeomagnetic experiments performed for grey limestone and red beds from Devonian sedimentary sequence in Podolia reveal Permian remagnetization corresponding to the pole position: 47°S/351.5°E for red beds and 45°S/340°E for grey limestone and the primary NRM component corresponding to the Devonian pole position (2.3°S/338.4°E for red beds and 0°S/329°E for grey limestone. The primary origin of Devonian component was based on the fact that it was recognized in different sediments—limestone and sandstone and carried by different magnetic minerals: MD magnetite in grey limestone, and detritic grains composed of primary hematite with ilmenite intergrowths in red beds. The fabric of AMS of red sandstone and these samples of grey limestone that preserved Devonian magnetization are typical for sedimentary structure.

The preservation of primary component of natural remanence was supported by analysis of mechanism of remagnetization: chemical in the case of red beds and thermo-viscous in the case of grey limestone.

Rock magnetic experiments revealed that in grey limestone the main carrier of NRM is MD magnetite. In red beds five forms of hematite were present: detritic grains of hematite with ilmenite intergrowths, martite, crystals of specular hematite within chlorite grains and two forms of hematite pigment. The first three forms of hematite have unblocking temperature of about 690 °C and could be the carriers of detrital or early diagenetic remanence. Pigmentary hematite has T_{ub} of approximately 590–600 °C and carries secondary component. Hysteresis properties suggested that red beds underwent chemical remagnetization whereas grey limestone are not altered.

The remagnetization pattern is in agreement with the thermal history of Lower Palaeozoic rocks from Podolia. Maximum palaeotemperatures during burial reached 200 °C. The age of maximum palaeotemperature is about 300 Ma, in excellent agreement with the position of secondary poles on APWP.

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