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DATA REPOSITORY

METHODS

Determination and quantification of whole rock samples by XRD

The mineral characterization and quantification were done on whole rock and fine fraction samples on random oriented powder mounts and textured glass slides using the Panalytical- Empyrean X-ray diffractometer (XRD, installed at the University of Geneva). The analyses were performed in continuous scan mode using Bragg-Brentano geometry with a step size of 0.013°2Θ/step and a counting time/step of 350 s in the range from 4 to 70° (45 kV, 40 mA, Cu K-α). The preparation, sample treatment and analysis of the XRD-pattern were carried out according to the recommendations by (Moore and Reynolds, 1997). Detection limit for qualitative mineral identification is around 5 wt%. The mineral determination was performed using the software HighScore Plus 3.0e.

Determination of the illite crystallinity or Kübler Index (KI)

The measurement of the illite crystallinity was performed on glass plates, according to (Weber, 1972) recommendation to prepare "thin" texture compounds using 1.5–2.5 mg/cm^2. The samples were scanned from 6 to 11 °2Θ, using a step size of 0.0131°2Θ and a measuring time of 400 s/step in continuous mode. The illite crystallinity was determined using the software IDEFIX developed at the Geosciences Center of the University of Göttingen (Friedrich, 1991). KI values may theoretically range from 0.060 Δ°2Θ (ideally ordered muscovite) to 1 Δ°2 Θ (poorly ordered, I/S mixed layers). The KI values were calibrated against the CIS standards introduced by [Warr and Rice, 1994]) (Equations 1 and 2). The calibration was done following the procedure of (Doublier et al., 2012). KI_{CIS} corresponds to the Kübler Index correlated against CIS standards, KI_m corresponds to the measured Kübler Index and $KI_{Kü}$ to the Kübler Index limits given in Kübler (1964), respectively.

$KI_{CIS} = 0.7507*KI_m+0.1366$ ($R^2 = 0.873$) (1)

$KI_{Kü} = 0.8221*KI_{CIS}+0.015$ (2)

Illite 10 Å peak decomposition

The illite peak decomposition and quantification of illite phases was performed using the program Newmod© (Reynolds Jr. and Reynolds III., 1996). All measurements were performed in the glycolated state.

Polytype quantification

A total number of 40 (including different grain size fractions) illite polytype quantification of 23 samples was performed to determine the relative amount of detrital/metamorphic $2M_1$ illite to diagenetic 1M illite on random powder mounts using the sideload-method described and recommended in Moore and Reynolds (1997) and Grathoff et al. (1998). The samples were analyzed from 16 to 44 °2Θ using a step size of 0.0131 °2Θ and a counting time of 400 s/step in continuous mode. The polytypes are quantified by measuring the area/intensity of their specific peaks (in case of $2M_1$ of five specific peaks) and rationing them against the area/intensity of a peak at 2.58 Å, which is common for both polytypes (Grathoff and Moore, 1996).

K/Ar geochronology

Fundamental concept of K/Ar dating and detrital age calculation

K/Ar analysis. The age determination analysis consists of two distinct analyses, the determination of K_2O and of the isotopic Ar content. The K_2O content was analyzed in duplicates using a BWB XP flame photometer. The samples were dissolved in a mixture of HF and HNO_3 followed by adding a CsCl suspension (12.5%) as an internal standard and ionization buffer for alkali metals, respectively. The pooled error of the duplicate potassium determinations in samples and standards is better than 1%. For Ar-analysis, the samples were pre-heated at high vacuum at 120 °C for at least 24h to reduce the amount of atmospheric argon attached to the mineral surfaces. The argon isotopic composition was analyzed in a Pyrex glass extraction and purification line coupled to an ARGUS VI multi collector mass spectrometer operating in static mode. The amount of radiogenic ^{40}Ar was determined by isotope dilution method using a highly enriched ^{38}Ar spike, which was calibrated against the international biotite standard HD-B1 (Fuhrmann et al., 1987). The extracted gases were purified in a multistage process by Ti- and SORB-ACs getters. The reproducibility of the Ar isotopic results was controlled by repetitive analysis of the Standard HD-B1. The obtained values were considered to be satisfactory and no corrections were applied to the raw data. The K/Ar ages were calculated with the usual decay constants (Steiger and Jäger, 1977), and the overall error is <1% (2σ).

SEM and TEM studies

A JEOL JSM7001F SEM equipped with the EDS system JED 2300 was used at the University of Geneva to characterize the textural relationship of rock components and the various clay mineral assemblages based on their textural intergrowth, semiquantitative chemical composition, crystal shape and size. The semiquantitative determination of the composition of the clay minerals served as a powerful tool to distinguish different clay mineral generations.

The JEOL 2100 TEM equipped with a JEOL energy dispersive spectrometry (EDS) system was used at SUVA Lucerne. The microscope was operated at 200 kV. Selected Area Electron Diffraction (SAED) and high resolution images were interpreted with the help of (J)EMS software (Stadelmann, 1987). The TEM was used as imaging device and to determine illite polytypes. A few mg of the samples 14–1 (<2 μm fraction) and 15–21 (<0.2 μm fraction) were suspended in isopropanol and a drop of the suspension deposited on a carbon-coated copper grid.

REFERENCES CITED

Doublier, M.P., Potel, S., Franke, W., and Roache, T., 2012, Very low-grade metamorphism of Rheno-Hercynian allochthons (Variscides, Germany): facts and tectonic consequences: International Journal of Earth Sciences, v. 101, no. 5, p. 1229–1252, doi:https://doi.org/10.1007/s00531-011-0718-3.

Friedrich, D., 1991, Eine neue Methode zur Bestimmung der Illit- Kristallinität mit Hilfe digitaler Messwerterfassung. [Diploma thesis], Georg-August-Universität Göttingen.

Fuhrmann, U., Lippold, H.J., and Hess, J.C., 1987, Examination of some proposed K–Ar standards: 40Ar/39Ar analyses and conventional K–Ar data: Chemical Geology, v. 66, p. 41–51, doi:10.1016/0168-9622(87)90027-3.

Grathoff, G.H., and Moore, D.M., 1996, Illite polytype quantification using Wildfire© calculated X-ray diffraction patterns: Clays and Clay Minerals, v. 44, no. 6, p. 835–842, https://doi.org/10.1346/CCMN.1996.0440615.

Grathoff, G.H., Moore, D.M., Hay, R.L., and Wemmer, K., 1998, Illite polytype quantification and K–Ar dating of paleozoic shales: a technique to quantify, *in* Schieber, S., Zimmerle, W., et al., eds., Shales and Mudstones, Vol. II: Petrography, Petrophysics, Geochemistry, and Economic Geolology: Stuttgart, E. Schweizerbart'sche Verlagsbuchhandlung, p. 161–175.

Kübler, B., 1964, Les argiles, indicateurs de métamorphisme: Revue de l'Institut Français du Pétrole, v. 19, no. 10, p. 1093–1112.

Moore, D.M., and Reynolds, R.C., Jr., 1997, X-ray Diffraction and the Identification and Analysis of Clay Minerals: Oxford, Oxford University Press.

Reynolds, R.C., Jr., and Reynolds, R.C., III, 1996, NEWMOD-for-Windows: The Calculation of One-Dimensional X-ray Diffraction Patterns of: Hanover, New Hampshire.

Stadelmann, P.A., 1987, EMS - a software package for electron diffraction analysis and HREM image simulation in materials science: Ultramicroscopy, v. 21, no. 2, p. 131–145, doi:https://doi.org/10.1016/0304-3991(87)90080-5.

Steiger, R.H., and Jäger, E., 1977, Subcommission on Geochronology: Convention on the Use of Decay Constants in Geo- and Cosmochronology: Earth and Planetary Science Letters, v. 36, p. 359–362, doi:https://doi.org/10.1016/0012-821X(77)90060-7.

Warr, L.N., and Rice, H.N., 1994, Interlaboratory standardization and calibration of clay mineral crystallinity and crystallite size data: Journal of Metamorphic Geology, no. 12, p. 141–152, https://doi.org/10.1111/j.1525-1314.1994.tb00010.x.

Weber, K., 1972, Notes on determination of illite crystallinity: Neues Jahrbuch für Mineralogie Monatshefte, v. 6, p. 267–276.