# Estimates of paleostress and paleotemperature conditions from naturally deformed calcite grain aggregates: Examples from Inner Western Carpathians, Slovakia

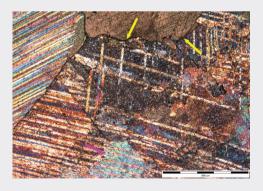
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Abstract: Twin lamellae in calcite rocks or veins represent an important tool for determination of paleostress and paleotemperature conditions. To determine the values of paleostress in MPa, registered by the deformed monominerallic calcite rock or vein and expressed as differential stress ( $\sigma_D = \sigma_1 - \sigma_3$ ), we used two paleopiezometric methods: Method of Twinning Incidence ( $I_t$  = twinned grains/total number of grains) and Twin Density ( $D = \sigma_1 - \sigma_2$ ) number of twins to 1 mm of the grain size perpendicular to twins). Measurements were done on marbles of the Bôrka Nappe (exhumed relics of the Meliata-Hallsttat Ocean) and calcite veins present in the Pieniny Klippen Belt (inferred suture zone after the Penninic Ocean). Presented paleopiezometric results document the differential stresses reached during the Meliata Unit Bôrka Nappe marble exhumation and thrusting over the Gemericum after the closure of the Meliata-Hallstatt Ocean. Whereas the deformed calcite veins from the Pieniny Klippen Belt, as a product of the last deformation event, provide the differential stresses related to the final deformation of the host rocks. Results of meaurements have proved the increase of the mean width of calcite twins (t - twin-width) by prograding temperature (T). The low twin width in marbles ( $t = 0.8-1 \mu m$ ) was caused by deformation at low paleotemperature conditions (T = 170–200 °C). Compared to this, the calcite veins with twin width 9.4–15.9  $\mu$ m indicate much higher paleotemperature conditions during their deformation (T > 250 °C). The grain-size of calcite crystals is decreasing with increasing differential stresses  $(\sigma_p)$ , whereas the number of twins per 1 mm of perpendicular size of the grains (D) is increasing. The small grain-size in marbles ( $d = 23.7-58.2 \mu m$ ) and high number of twins/1 mm (D = 73–646) is consequenced by high differential stresses ( $\sigma_D$  = 389.37–429.55 MPa). Opposing to this, the high grain-sizes in calcite veins (d = 450.5–516 µm) and low number of twins/1 mm of perpendicular size of the grains (D = 31–52) indicate lower differential stresses ( $\sigma_D$  = 203.48–240.44 MPa). The twin-length (l) is strongly dependent on the grain diameter (d) and represents a key parameter at calculations of the Volume of twins ( $V_{twins}$ ). The low value of twin-length (1 = 14.27–38.675 µm) in marbles is caused by the low grain-size (d = 23.7–58.2 µm), what is in contrast with results from calcite veins, where the high value of twin-length (1 = 77.08.973.25 µm) was caused by the low grain-size (d = 450.5–516 µm).  $(1 = 770.8 - 972.35 \,\mu\text{m})$  was caused by the big grain-size  $(d = 450.5 - 516 \,\mu\text{m})$ . Due to low value of twin-length in marbles the volume of twins  $(V_{\text{twins}} = 37.7 * 10^3 - 251.9 * 10^3)$  is much lower in comparison to volume of twins in calcite veins  $(V_{\text{twins}} = 9.9 * 10^6 - 410.7 * 10^6)$ , having the bigger twin-length.

**Key words:** paleopiezometry, differential stress, calcite grains, Twin Density, Twinning Incidence, Inner Western Carpathians

Graphical abstract



inights

- The principal parameters of the twinned grains in calcitic rocks are reviewed in relation to numerically revealed differential stresses
- Differential stress σ<sub>D</sub> = σ<sub>1</sub> σ<sub>3</sub> (MPa) represents a numeric value suitable for comparison of the strain in individual parts of the shear zone and such providing data for revealing the deformation softening and hardening, respectively.

#### 1 Introduction

Nowadays numerous investigations deal with the rheological and mechanical behavior of rocks during their deformation. The strain of rocks is a result of a stress magnitudes, acting during deformation at a temperature. This paper focusses on mechanical twining on e-planes, which is a typical feature of naturally deformed calcitic rocks. It studies the relationship among individual parameters, including the computation of differential stress ( $\sigma_D = \sigma_1 - \sigma_3$ ; MPa). The differential stress is a value usable for comparison of the strain mechanisms e.g. in individual parts of the shear zone (such as deformation hardening or softening), although it does not provide information about spatial orientation of individual stress axes.

The deformation process in the calcitic rock (coarse-grained limestone, marble or calcite-vein deformation) via the calcite crystals deformation is the stress and temperature-dependent, so it can roughly document also the metamorphic conditions during deformation if the differential stress numerically expressed. The works of earlier authors report the deformation characteristics for T < 300 °C (Barber & Wenk, 1979), or even T < 400 °C (Turner, 1953; Carter and Raleigh, 1969; Groshong, 1988).

Within the Western Carpathians, paleopiezometric research (Németh, 2005; Németh et al., 2012; Zákršmidová et al., 2016) was focussed on two principal tectonic zones:

The 1<sup>st</sup> group of paleopiezometric measurements related to the Middle-Late Jurassic closure of the Neotethyan (Meliata-Hallsttat) Ocean and its elements, forming Jurassic Mélange (cf. Krystyn & Lein in Haas et al., 1995), encompassing also the Western Carpathian Bôrka nappe (Mello et al., 1998; Aubrecht et al., 2010), which were subducted and subsequently thrust over the Gemeric Unit within the Inner Western Carpathians – IWC.

The 2<sup>nd</sup> group of paleopiezometric measurements was focussed on the Pieniny Klippen Belt (PKB), representing ca. 600-700 km long geosuture following the boundary of Inner/Outer Carpathians (e.g. Andrusov, 1965; Scheibner, 1968; Mahel', 1981; Birkenmajer, 1976a, 1986; Nemčok et al., 1998 in Jurewicz 2005). The name of the PKB is associated with the existence of two branches of Penninic Ocean, the South Penninic – Vahic Ocean and the North Penninic – Magura Ocean (Plašienka et al., 1997). The final ocean closure occurred no earlier than the Early Eocene (e.g. Oberhauser, 1991, in Ratschbacher et al., 2004).

#### Dependence of calcite twins on paleostresses

Paleostresses are expressed in exact numerical values as differential stresses ( $\sigma_D$ ). They can be determined by two different methods (Rowe and Rutter, 1990): Twinning Incidence method ( $I_t$  = twinned grains/grains total) and Twin Density method (D = twins/length), both in MPa. Another type of measurements takes into calculation the Volume of fraction of twins ( $V_T$  = volume of twins/total volume), giving us the results in  $\mu m^3$ . This method is indirectly associated with previously mentioned piezometers.

The paleopiezometric measurements are based on the approaches by Jamison and Spang (1976), Lacombe (2007) and Laurent et al. (2000), who assumed that CRSS – Critical resolved shear stress ( $\tau_c$ ) is constant – about 10 MPa.

According to Burkhard (1993) the deformation twins are developed at very low shear stress varying in between  $\tau_c$  = 2–12 MPa, what is depending on temperature, as well as pressure conditions. The stress required to activate twinning is likely varying with the shear modulus, decreases with temperature (Rowe and Rutter, 1990). Twinning of calcite is a strain-hardening process, meaning that further twinning is resisted as beds tilt during subsequent deformation.

#### Dependence of calcite twins on paleotemperatures

The size of calcite grains increases with the increase of temperatues, leading to grain-boundary area reduction – GBAR mechanism, as a consequence of a static recrystallization – SR (Passchier and Trouw, 1996).

Not only the grain-size diameter, but also the width of calcite twins depends on paleotemperature. The morphology of calcite twins has been proposed as a geothermometer for low-grade metamorphic conditions (Burkhard, 1993; Ferrill, 1991; Ferrill et al., 2004).

The calcite twins deformed at temperature bellow T<170 °C are thin, what is visible as thin black lines with their typical width less than 1  $\mu m$  (Ferrill et al., 2004). Based on experimental deformation of calcite at higher temperature – T  $\geq$  300 °C done by Ferrill et al. (2004), the thickness of individual twins may exceed 5  $\mu m$ . Groshong (1974) suggested characteristic working definition of thin vs. thick calcite twins, pointed out their variable thickness strongly dependent on temperatures.

Burkhard (1993) and Ferrill et al. (2004) proposed following temperature dependent classification of twins: Type I – thin (<1  $\mu$ m) and straight twins (T = 170–200 °C); Type II – thick (>1–5  $\mu$ m) and slightly lensoidal twins (T = 150–300 °C), Type III – thick, curved and tapered twins, possibly re-twinned, accompanied by substantial dislocation glide (T > 200 °C), Type IV – thick and irregular twins, accompanied by a grain boundary migration (GBAR), indicating T > 250 °C.

In our research we have followed the previous studies, summarised in Németh (2005), Németh et al. (2012) and Zákršmidová et al. (2016), in which important parameters from paleopiezometric calculations have not yet been compared to each other. Paleostress and paleotemperature condidions represent the main factors influencing the shape, number and distribution of twins. We evaluate and compare the results from both suture zones – the Meliata Oceanic suture and the Penninic one.

#### 2 Research method

#### 2.1 Microscopic analytical techniques on calcite twins

Performed microscopic research of naturally deformed samples was based on calcite twin analysis. Studied samples were taken from the Meliatic fragments exposed

on Gemeric Unit (Inner Western Carpathians), as well as from the Pieniny Klippen Belt – representing the boundary between the Inner and Outer Western Carpathians.

Careful preparation of thin sections excluded the fracturing or twinning grains during the sample preparation. Examined thin sections were then optically analysed by optical microscope Olympus VX 53, having attached the high-resolution digital camera LUMENERA. Using methods of calcite paleopiezometry (discribed in chapter 2.2) we measured 240 calcite grains in each sample to determine differential stresses.

Within each twin set we also measured: the average twin-width – t, number of twins per 1 mm of the perpendicular size of the grain – D, as well as grain-size – d. Twins, microfractures and pressure solution seams were present in large calcite grains, but the intense twinnig was regarded as the dominant deformation mechanism.

We quantified the density of calcite twins, as well as the percentage of grains with optically visible deformation twins using two calcite piezometers – D (Twinn Density) and  $I_{\iota}$  (Twinning Incidence) to obtain an estimate of the paleo-stresses governing deformation (MPa). We also determined the total volume of twins  $V_{\iota}$  (Volume of twins/total volume). The average twin lamellae width was used for paleotemperature ( ${}^{\circ}C$ ) estimations.

#### 2.2 Differential stresses determination

Currently it is generally known that calcite twins represent an important tool to determine the differential stresses (Blenkinsop, 2000; de Bresser and Spiers, 1997; Masuda et al., 2011; Molli et al., 2011; Passchier and Trouw, 1996; Rybacki et al., 2011, 2013; Tullis, 1980; Turner, 1953; Wenk et al., 1983, 2006).

In our study, following previous studies by Rybacki et al. (2011, 2013), we focused on calcite paleopiezometry, being best calibrated for monomineralic calcite marbles with a pervasive ductile deformation (Rowe & Rutter 1990), as well as calcite veins which penetrated the whole volume of host rocks.

Calcite paleopiezometry is based on the size of dynamically recrystallized grains and the number and character of deformation twins. There are known two independent ways how to determine differential stresses in calcite rocks: Twinning Incidence (It) and Twin Density (D).

Twinning incidence, I, is defined as the percentage of grains that demonstrates microscopically visible twins. During observation in thin section there were visible 1 or 2 twin sets.

The differential stress  $\sigma_D$  ( $\sigma_1 - \sigma_3$ ; MPa) can be distinguished by the equation below, where d represents the size of grains in  $\mu$ m. The standard error in this technique, as stated by Rowe & Rutter (1990), is up to 31 MPa.

$$\sigma_{\rm p} = 523 + 2.13 \text{ L} - 204 \log d \text{ [MPa]}$$

Twin density, D, is defined as the number of twins regarding the grain diameter, measured perpendicularly to the twins. To determine the twin density, the number of twins perpendicular to the twin boundaries within individual grains was counted and normalized to a unit length of 1 mm.

As shown by Friedman and Heard (1974), as well as Rybacki et al. (2013), twin density measurements using a flat stage match those measured with the universal stage to within 10 %. For maximum representativeness of provided data we performed twin density measurements at more than 3 different position to reach the maximum correctness. The standard error in this method is 43 MPa.

The relation of the differential stress on twin density D is as follows:

$$\sigma_{\rm D} = -52.0 + 171.1 \log D \text{ [MPa]}$$

The primary data were obtained from thin sections, measuring the grain size in  $\mu$ m, as well as the number of deformation twins in corresponding grains. To guarantee the maximum representativeness of data, measurements were done systematically on profiles through the thin section, from grain to grain. Extreme dimensions (extremely small or large grains) were excluded from following calculations, using the variation coefficient 0.25, defined by Ranalli (1984). For numerical processing, a procedure consisting of several steps was developed (see in Németh, 2005).

Another type of piezometer is based on obtaining the average volume of all twins –  $V_{twins}$  = volume of twins/total volume ( $\mu$ m³). The total fractional twin volume –  $V_T$  is the most important (Ferrill et al., 2004; Groshong, 1972; Chapter in Kocks et al., 1998), what equals to  $N_V$  – twins per unit volume of rock, as well as to  $V_{twins}$  – volume of all twins (Ferrill et al., 2004; Groshong et al., 1984 in Rybacki et al., 2013).

$$V_{T=}N_{V=}V_{twin}$$

For a randomly oriented oblate spheroids with their average length l, width t, and aspect ratio q = t/l, there were developed the below stated equations (Barnett et al., 2012; Dehoff and Rhines, 1961; Underwood, 1970 in Rybacki et al., 2013), using correlation between the product of twin density and twin volume (Groshong, 1972, 1974, and Groshong et al., 1984).

$$V_{twins} = \pi/6. \ D.q.l^3$$
,

where D represents number of twins/1mm; t – twin-width and l – twin-length, q = t/l.

### 2.3 Twin width measuring for paleotemperature estimation

Supposing that the width and shape of the twins mirror the paleotemperature conditions, we performed measurements focusing on these parameters.

Because of the twin widths can not be easily measured using a flat-stage, we realized measurements including many twins with random deep angle according to the previous researches done by Underwood (1970). For a set of twins dipping at random angles to the traces, we measured the average of the true distances between adjacent boundaries by the equation below, stated by Rybacki et al. (2013), where w ( $\mu m$ ) represents the average distances measured perpendicular to the traces of twins in thin section

$$w^* = 2/\pi \cdot w$$

Our calculations were realized according to Ferrill et al. (2004), in which the "mean twin width" for a sample was evaluated by first measuring of the average twin width for each twin set (twin set average) and by second averaging the twin-set averages for the sample. The average of each twin set is calculated by summing the width of twinned material composed of both thick and thin twins. This sum was finally divided by the total number of twins in the set.

#### 3 Results

The calcite twin data presented here are taken partly from previously published studies by Németh et al. (2012) and Zákršmidová et al. (2016). Recent study shows a noticable correlation of previously obtained results with new ones, which completed them.

The numeric estimations of differential stresses - paleostresses ( $\sigma_D$ , MPa) and paleotemperature (T, °C) were based on measurements of following parameters: d – grain-size, l – twin-length, t – twin-thickness, D – no. of twins per 1 mm. Calculation of differential stress was performed by two different methods of paleopiezometry: It – Twinning Incidence, D – Twin Density, as well as the method of determination of Vt – Volume of fractures of twins. We pointed out the mutual relationships between all obtained values.

The obtained results are divided into 2 data sets. The 1<sup>st</sup> one is represented by marbles and the 2<sup>nd</sup> by calcite veins penetrating the host rocks.

#### 1st set of data

The first set of samples (cf. Németh et al., 2012) contained six fine-grained limestones, representing five marbles of the Meliata melange nappe outlier with strong recrystallization (JAK-5, JAK-7, JAK-48, JAK-46, JAK-46).

Tab. 1

Twinning Incidence (It) and Twin Density (D) and calculated paleostress conditions in studied samples from the Kurtová skala hill – frontal part of the Bôrka nappe – "Jaklovce Meliaticum" (exhumed mélange of the Meliata-Hallsttat Ocean/Meliata Unit) thrust over the Gemeric Unit. The earlier laboratory paleopiezometry and computing, done by Zákršmidová (2012) in Németh et al. (2012), is completed by new results.

		1	winnin	g Incid	ence (It	Twin Density (D)			Twin Volume (Vt)			
	Representative grain- size - d	Calcul. without variation coef.	nined by ent	Calculation with variation coefficient			1 mm eter	Calculat.	Calculat.	e twin-	e twin-	Calculat.
		without variation coef.  Calcul. with variation coeflicient - for interval determined by a configuration coeflicient coefficient coefficie	Calculat. with the whole It	Arith. mean of σ for size interval classes	Weight. mean of σ for size classes	<b>D</b> - no. of twins per 1 m of perpend. diameter	without variation coeffic.	with variation coeffic.	Representative twin- length - I	Representative twin- thickness - t	of the whole Vt (volume of twins/total volume)	
	μm	σ (МРа)	±-	σ (МРа)	σ (МРа)	σ (МРа)		σ (МРа)	σ (МРа)	μm	μm	μm
Jak-5	23.7	269.40	12.22	268.72	272.07	269.40	173.05	330.95	347.49	38.675	0.8	136.2*10³
Jak-7	27.7	356.45	59.52	355.62	353.29	281.27	355.32	384.41	386.44	14.27	1.0	37.7*10³
Jak-46	40.06	411.63	100	407.79	405.79	408.52	481.61	407.93	406.73	19.65	1.0	98.7*10³
Jak-48	58.2	208.46	99.29	374.42	373.43	375.30	381.59	389.61	389.37	35.125	0.8	251.9*10 <sup>3</sup>
Jak-54	42.2	407.15	100	403.40	403.73	404.25	428.88	429.55	429.55	20.3	0.8	139.5*10³

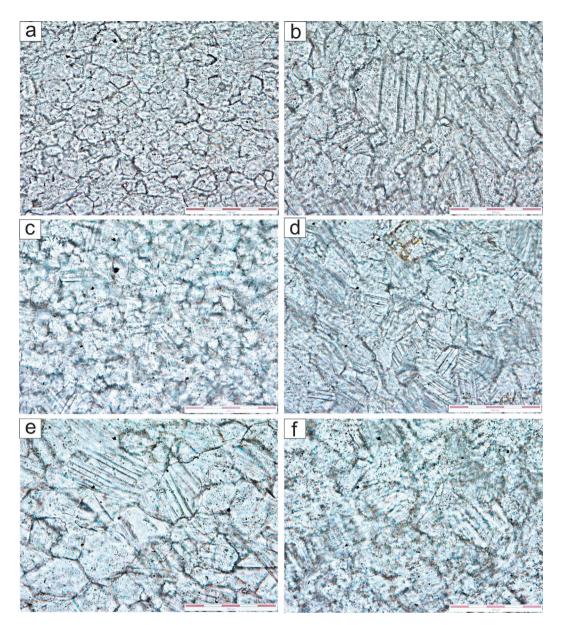


Fig. 1. Representative photomicrographs of undeformed (a) and deformed calcitic marbles (b-f) in plane-polarized light (PPL images), characterized by the single or multiple twin sets. The thin twins indicate the low-temperature (LT) conditions. **b-f** – The high-strain deformation microstructures demonstrated by the bent twins, as well as high differential stresses ( $\sigma_D$ ), Twinning Incidence (It) and Twin Density (D).

Other microscopic indicators, showing completely recrystallized calcite grains in marbles, except for the twinning, are as follows: boulging (BLG), subgrain rotation (SGR), grain boundary migration (GBM) and the grain boundary area reduction (GBAR).

a – JAK-4: Undeformed limestone, without microscopically visible twins, lying in autochthonous position beneath the deformed marbles (b-f) of the nappe outlier, transported by the kinematics of superficial nappe with remaining "frozen" high-strain state from the subduction zone (allochthonous body). Photomicrographs of studied samples are depicted with following parameters: d – average grain-size; l – twin-length; t – twin-thickness; It - Twinning Incidence – the percentage of grains with deformation twins; D – Twin Density – the average number of twins per 1 mm perpendicular to twin lamellae; σ<sub>D</sub> – differential stress (MPa). The scale bar in each case (a-f) represents 100 μm. Plane polarized light.

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b – JAK–5: d=23~\mu m; l=38.7~\mu m; t=0.8~\mu m; l=12.2; D=330.95; \sigma=347.49~MPa c – JAK–7: d=27.7~\mu m; l=14.3~\mu m; t=1.0~\mu m; lt=59.52; D=384.41; \sigma=386.44~MPa d – JAK–46: d=40.6~\mu m; l=19.7~\mu m; t=1.0~\mu m; l=100; D=407.93; \sigma=406.73~MPa e – JAK–48: d=58.2~\mu m; l=35.1~\mu m; t=0.8~\mu m; l=99.29; D=389.71; \sigma=389.37~MPa f – JAK–54: d=42.7~\mu m; l=20.3~\mu m; t=0.8~\mu m; l=100; l=428.88; l=20.55~MPa
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54) and one limestone (JAK-4) representing the autochthonous footwall without recrystallization, being excluded from the further research. The study contributed to deciphering of the boundary between autochthonous cover limestones and allochthonous body of so-called "Jaklovce Meliaticum", being exhumed from the Meliata-Hallstatt Ocean/Meliata Unit suture zone, located south of Gemericum, forming the Jurassic Mélange (e.g. Krystyn & Lein in Haas et al., 1995) and being transported over Gemericum (Dallmeyer et al., 1996; Putiš et al., 2011, 2014). The dynamic recrystallization was in studied thin-sections manifested also by bulging (BLG), subgrain rotation (SGR), grain boundary migration (GBM) or grain boundary area reduction (GBAR).

The stress field and recrystallization of the whole volume of calcite marbles caused the origin of deformation e-twins nearly in each calcite grain (Twinning Incidence –  $I_t$  up to 100 %; **Tab. 1, Fig. 1**). The twin densities (D) vary between 174 and 647 twins per 1 mm at a very small size of grains, ranging from 24 to 58 ( $\mu$ m). The calcite paleopiezometry revealed the differential stresses ( $\sigma_D$ ) within the range of 347.49-429.55 (MPa).

Other our measurements included the average thickness of twins (t), ranging from 0.8 to 1  $\mu m$  with the average spacing between them from 0.5 to 0.8  $\mu m$ . We established the low temperature deformation conditions less than 200 °C (sensu Burkhard, 1993) using obtained data, which suit to recrystallization in a subduction zone environment. The twin-thickness (t) represents an important parameter for calculations of the average volume of twins –  $V_{twins}$ . We have obtained results ranging from  $37.7*10^3$  to  $251.9*10^3 \, \mu m^3$ . We also measured a so-called "key parameter" – twin-length (l), which was in the range of 14.27 and 38.68  $\mu m$ .

#### 2nd set of data

The 2<sup>nd</sup> data set of thin-sections was compiled from representative samples from the Pieniny Klippen Belt (PKB), forming a boundary zone between the Inner Western Carpahians (IWC) and the Outer Western Carpathians (OWC). The sampling of a regional extent was focused on the interconnection zone between the older PKB shear system and younger Subtatric Ružbachy fault system (SRFS), which is localized in the eastern sector of this intricate belt system near the village of Chmel'nica.

We performed microscopic studies on specimens represented with twinned calcite veins (thick 5–15 cm) of regular shape, penetrating the whole volume of the host rocks – calcite grey marly limestones of Jurassic–Early Cretaceous age. In thin sections we distinguished more deformed older veins penetrated by less-deformed younger ones.

Within 240 measured calcite grains in each thin section (Fig. 2), we used the grain-size diameter (d) – varying from 450 to 516  $\mu$ m, as well as the number of deformation twins (D) with 31–52 twins per mm of perpendicular diameter. These parameters provided the differential stresses ( $\sigma_D$ ) ranging from 203.48 to 228.44 (MPa), as can be seen in Tab. 2.

We also observed indicators connected with manifestations such as bulging – BLG, subgrain rotation – SGR, grain boundary migration – GBM, as well as grain boundary area reduction – GBAR mechanisms, tightly associated with the onset of static recrystalization – SR (polygonization of calcite grains). All these indicators are related to ductile deformation (Passchier & Trouw, 1996).

The average volume of twins  $V_T$ , ranging from  $9.9*10^6$  to  $410*10^6\,\mu m^3$  is the result of the twin-length (l) 770.8-972.35  $\mu m$ , as well as twin-thickness (t) of  $9.43-15.9\,\mu m$ . The results of twin-thickness, as well as the shape of twins

Tab. 2
Twinning Incidence (It), Twin Density (D) and Twin Volume  $(V_T)$ , as well as differential stresses calculated for the calcite veins from the eastern sector of the PKB – Chmel'nica village.

		Twinning Incidence (It)					Twin Density (D)			Twin Volume (Vt)		
	Representative grain- size - d	Calcul. without variation coef.	ned by	Calculation with variation coefficient			1 mm ter			twin-	twin-	Calculat.
		Calcul. weight mean	- for interval	Calculat. with the whole It	Arith. mean of σ for size interval classes	Weight. mean of σ for size classes	<b>D</b> - no. of twins per 1 mr of perpend. diameter	Calculat. without variation coeffic.	Calculat. with variation coeffic.	Representative t length - I	Representative t	of the whole Vt (volume of twins/total volume)
	μm	σ (МРа)		σ (МРа)	σ (MPa)	σ (МРа)		σ (МРа)	σ (МРа)	μm	μm	μm
Jak-5	516	119.46	66	110.27	124.42	119.46	31	199.4	203.48	770.8	9.43	9.9*10³
Jak-7	450.5	201.97	36	199.78	226.2	201.97	52	242.6	240.44	972.35	15.9	410.7*10 <sup>3</sup>

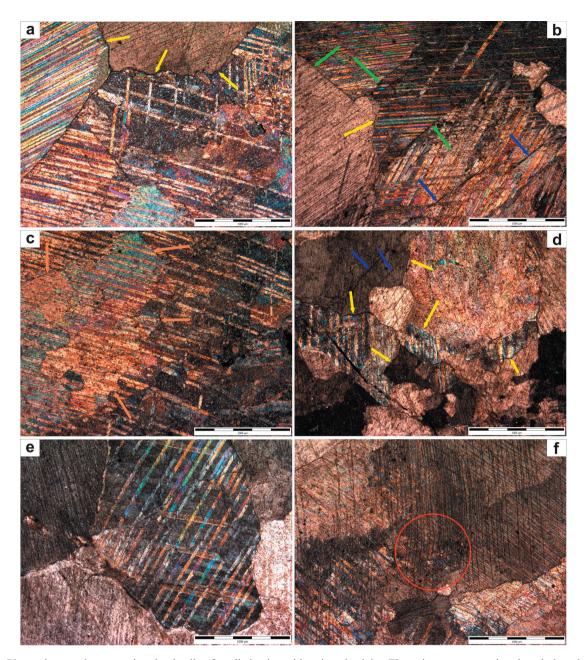


Fig. 2. Photomicrographs presenting the details of studied veins with twinned calcite. The veins are penetrating the whole volume of the Jurassic–Early Cretaceous marly limestone as a host rock. The Pieniny Klippen Belt, Western Carpathians.

Numerical expressions in the description below relate to following parameters: d – average grain-size, l – twin-length; t – twin-thickness; It - Twinning Incidence – the percentage of grains with deformation twins; D – Twin Density – the average number of twins per l mm perpendicular to twin lamellae;  $σ_D$  – differential stress (MPa). Photographs in crossed polarized light. Scale bar = 100 μm.

PKB3a:  $d = 516 \mu m$ ;  $l = 770.8 \mu m$ ;  $t = 9.4 \mu m$ ; lt = 66; D = 199.4;  $\sigma = 203.48$  MPa (a, c, e) PKB3b:  $d = 91 \mu m$ ;  $l = 244.4 \mu m$ ;  $t = 15.9 \mu m$ ; lt = 36; D = 242.6;  $\sigma = 240.44$  MPa (b, d, f)

The deformed calcite veins in cross-polarized light (XPL images) are characterized by the single or multiple twin sets, developed mostly at different angles. The coarse-grained calcite in the vein manifest high twin density. The deformed calcite grains are slightly elongated or sub-isometric. The internal strain is achieved under the thin-thick-twin regime, which indicates the low-temperature conditions. Scale bar represents  $1000 \, \mu m$ . a – deformation-induced textural re-equilibration in ductile / brittle-ductile transition regime; note grain-boundary bulging – BLG (yellow arrows; as well as in b-d); b – static recrystallization accompanied by grain boundary area reduction – GBAR manifested by polygonization (straight boundaries between calcite grains; green arrows). Microfractures are shown by blue arrows. c – Irregularly shaped calcite grains show the evidence of grain-boundary migration with forming of subgrains; d – bulging (yellow arrows); microfractures (blue arrows) and misoriented grain (pale-coloured grain close to the centre); e – large calcite grain with two sets of twins; f – bending of twin planes at high differential stresses (red circle). Bent thick twinning lamellae are produced during decompression (Pieri et al., 2001). Note twin lamellae cross-cuting host calcite crystals as in the case e.

point out on **low-temperature twinning conditions (below 200** °C *sensu* Burkhard, 1993).

#### 4 Interpretation and discussion

Twinning in calcite represents an important deformation mechanism of low-temperature conditions (150–300 °C) and it is leading mechanism during the high-pressure conditions (Groshong, 1972, 1988; Christian and Mahajan, 1995; Burckhard, 1993; Ferrill et al., 2004; Turner, 1964). Twinning begins at stress concentration on calcite crystals surfaces, grain boundaries, or other defects (Burkhard, 1993; Christian and Mahajan, 1995; Nicolas and Poirier, 1976).

We applied paleopiezometric measurements using two independent methods, such as Twinning Incidence  $(I_t)$  and Twin Density (D).

*In the 1<sup>st</sup> data set* (marbles), the revealed differential stresses were within the range of **347.49–429.55 MPa** (Németh et al., 2012).

These results together with previous researches (cf. Németh, 2005) have confirmed the allochthonous position of the Meliata (the Bôrka nappe) fragments in the North-Gemeric zone. Studied limestones, recrystallized due to subduction and subsequent exhumation represent a part of the Meliata-Hallsttat Jurassic Mélange (e.g. Krystyn & Lein in Haas et al., 1995). Presented results of our study in this topic are compatible with the research outcomes of Mello et al. (1998a, b); Mello et al. (1997); Ivan (2002); Ivan et al. (2009); Ivan & Méres (2009) or Putiš et al. (2011, 2012, 2014).

The 2<sup>nd</sup> data set presents the differential stresses within the range 203.48-240.44 MPa, revealed from the calcite veins, penetrating the folded beds. The research was focussed on the interconnection of the older PKB tectonic system and younger Sub-Tatra Mts. Ružbachy fault system (SRFS), located in the eastern sector of PKB system. In the studied outcrop near the Chmelnica village, we identified three deformational phases. The studied calcite veins originated during second deformation phase from fluids migrating throughout the east-west trending joints and being ductilely overprinted during third deformation phase related to shearing along the Ružbachy fault system (Zákršmidová et al., 2016).

According to earlier structural researches, the entire deformatiom of the PKB units virtually occured under very low temperatures at diagenetic conditions, which is accompanied by brittle structures, followed by the diffusion mass transfer mechanisms, such as the pressure solution and precipitation (Plašienka, 2012). The intricate structure of the PKB exhibits the manifestation of brittle, as well as ductile deformation due to polyphase tectonic evolution (e.g., Nemčok, M. & Nemčok, J.,1994; Jurewicz, 2005; Plašienka and Mikluš, 2010; Plašienka, 2012).

By means of high differential stresses accomodated by calcite grains (in limestones, or veins), leading to origin of ductilely deformed calcite crystals within examined samples, we have established empirical relationships among the most principal parameters, included into piezometric calculations, such as:  $d(\mu m) - calcite \ grain-size, \ D - no. \ of twins per 1mm of the grain size, perpendicular to the grain, <math>l(\mu m) - twin-length, \ t(\mu m) - the \ average \ twin-thickness.$ 

#### 4.1 Calcite grain-size – d (µm)

Twinning Incidence (I) vs. calcite grain-size (d)

Twinning Incidence (It) piezometer is based on a percentage of twinned grains regarding the whole number of grains. It provides results, which are independent on complexity of deformation history and orientation of the applied stresses (Rowe and Rutter, 1990). The differential stress revealed by the Twinning Incidence (It) piezometric method:

$$\sigma_D = 523 + 2.13 I_t - 204 \log d$$
 [MPa],

where  $I_t$  – Twinning Incidence, showing a percentage of grains with microscopically visible deformation twins and d – grain-size, the average diameter of grains.

The grain-size diameter decreases significantly with prograding deformation (mylonitization), and by this way also by increasing differential stress ( $\sigma_D$ ). The low grain-sizes in marbles (23.7–58.2 µm) were produced by high differential stresses, which was numerically confirmed –  $\sigma_D$  = 347.49–429.55 MPa, as can be seen in **Fig. 3a.** On the contrary, the larger grain-sizes in calcite veins (450.5–516 µm) reflect the lower differential stresses  $\sigma_D$  = 203.48–240.44 MPa and higher temperature static recrystalization (cf. Passchier and Trouw, 1996), being depicted in **Fig. 3b.** 

## 4.2 No. of twins per 1 mm of the perpendicular grain size (D)

Twin Density (D) vs no. of twins/mm

Differential stresses determined by the *Twin Density* (D) method are calculated applying the equation  $-\sigma_D$  = -52.0 + 171.1 log D [MPa], where D – the number of twins/1mm.

In all samples the *no. of twins per 1 mm (D)* increased by prograding differential stresses  $(\sigma_{D_i} MPa)$ , determined by *Twin Density (D)* paleopiezometry.

The high number of twins per mm (173.05–646.41) in marbles was caused due to high differential stresses –  $\sigma_D$  = 347.49–429.55 MPa.

Contrary to this in the calcite grains within veins is the number of twins per mm ranging between 31 and 52, which is much lower in comparison to marbles. It is due to lower differential stresses –  $\sigma_D = 203.48-240.44$  MPa.

#### 4.3 Twin-length – l (µm)

Volume of twins vs. twin-length

As was desribed before, the strain in rocks, accomodated by twins are expressed by several ways. One of them is also the total fractional twin volume, which is produced during deformation (Ferill et al., 2004; Groshong, 1972; Kocks et al., 1998).

Using the equation  $-V_{twins} = \pi/6$ .  $D.q.l^3$  ( $\mu m^3$ ), where D – no. of twins/1mm, q = t/l, t – twin-thickness, l – twin-length, we obtained new data about the volume fracture of twins ( $V_{twins} = \text{volume of twins/total volume}$ ).

fracture of twins ( $V_{twins}$  = volume of twins/total volume). The result of  $V_{twins}$  (Volume of twins) is strongly depended on twin-lengths (1), which represent a so-called

key parameter in calculation of  $V_{twins}$ . From the above stated equation it is supposed the linear relationship between  $V_{twins}$  and  $I_{twins}$ . On the basis of our measurements, it is evidenced that the low value of twin-length (14.27–38.68  $\mu$ m) in marbles caused lower values of  $V_{twins}$  ranging from 37.7\*10³ to 251.9\*10³ ( $\mu$ m³) in comparison to values yielded from calcite grains in veins, where twin-length in the range of 770.8 and 972.35 ( $\mu$ m) caused  $V_{twins}$  ranging from 99\*10³ to 410\*10³ ( $\mu$ m³). The relations between measured values can be observable in the **Fig. 5 a, b.** 

Twin-length vs. grain-size

The average twin-length (l) depends largerly on the grain-size, by which is the twin-length positively correlated. Lower value of twin-length (l) in the range of 14.27 and 38.68 ( $\mu$ m) is influenced by smaller grain-sizes parameters (23.7–58.2 $\mu$ m) in marbles, whereas in calcite veins are higher values of twin-length (9.43–15.6  $\mu$ m) caused

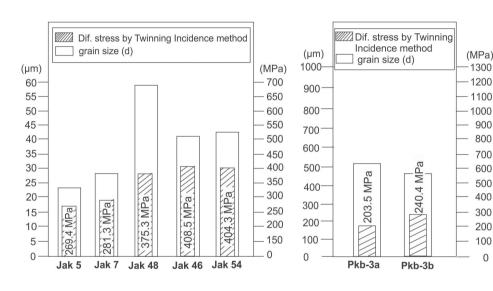
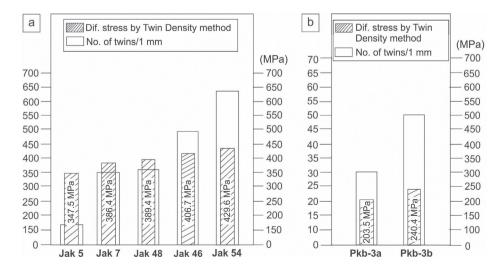


Fig. 3. a – The positive relationship between the differential stresses obtained by the Twinning Incidence method ( $\sigma_D$ , MPa) and grain-sizes – d ( $\mu$ m), except of the specimen – JAK-54 caused by SR – static recrystallization b – The negative relationship between Twinning Incidence – It ( $\sigma_D$ , MPa) and grain-sizes – d ( $\mu$ m) in comparison to marbles in a, caused due to higher temperature related static recrystallization.



**Fig. 4. a** – The positive relationship between differential stress determined by the Twin Density method (MPa) and number of twins per 1 mm (D) in the 1<sup>st</sup> data set (marbles). **b** – The positive relationship between differential stress determined by the Twin Density method (D, MPa) and number of twins per 1 mm (D) in the 2<sup>nd</sup> data set (calcite veins).

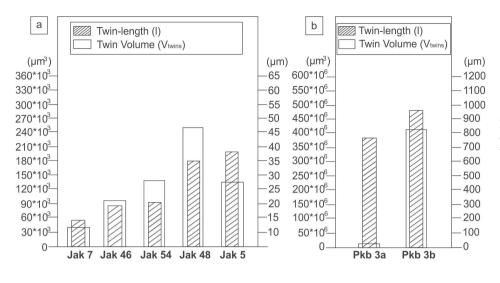


Fig. 5. The relationship between Volume of twins - V<sub>twins</sub> and twin-length - 1.

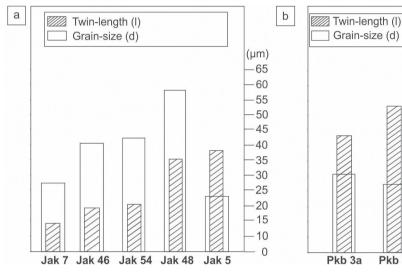


Fig. 6. The relationship between twin-length – l and grain-size – d.

due to larger grain-sizes (450.5–516 µm), as can be seen in Fig. 6.

#### 4.4 The average twin-thickness (µm)

To determine the average volume of twins (Vt), we studied characteristic indicators of twins for each grain-size interval (d), among which belong the average thickness of twins (t), as well as their average twin-length (l). We followed previous observations in naturally strained carbonates (Burkhard, 1993; Ferrill et al., 2004 and Groshong et al., 1984). The all those researches showed that thick calcite twins predominate above 200°C, while calcite twins formed below 200°C are thinner.

The average thickness of twins in marbles was 1 µm with almost uniform spacing between them (ranging from 0.5 to  $0.8 \mu m$ ), what mirrors low temperature – LT conditions (T< 200°C). Calcite grains within veins were thicker (up to 15.9 µm) with spacing between them, varying from 7 to 15 μm, as well as with manifistations of GBAR – grain boundary area reduction mechanism. It was consequenced due to higher temperatures (T > 250  $^{\circ}$ C).

(µm)

1200

- 1100

1000

900

800

700 600

500

400

300

200 100

0

Pkb 3b

The paleo-stress measurements realized within calcite crystal aggregates in marbles may approximate the peak metamorphic conditions. Those data from calcite veins, penetrating the whole volume of host rocks, may indicate a younger tectono-thermal reactivation from the final deformation stage.

#### 5 Conclusion

Twins in calcite grains represent appropriate paleostress (differential stress– $\sigma_{\rm p}$ ) and paleotemperature indicator for deformed calcitic rocks. Twinning piezometers according to Rowe & Rutter (1990) were used to determine the differential stress conditions during deformation of Meliata calcite marbles undergoing the subduction, exhumation and nappe displacement (the Bôrka nappe).

The multi-variant numerical processing followed the procedure by Németh (2005). The visualizations in this paper intend to present relationships between individual parameters included into paleopiezometric calculations: d - grain-size, D - number of twins per 1mm of the perpendicular dimension of the grain, 1 – twin-length and t – twin-thickness. As our research confirmed, the increasing number of deformation twins in marbles (D = 173–646) relates to increasing differential stresses ( $\sigma_p =$ 347.49–429.55 MPa). The lower number of deformation twins in calcite veins (D = 31-52) is resulting from lower values of differential stresses ( $\sigma_{\rm D} = 203.48-240.44$  MPa). The calcite grains in studied marbles had lower grain-size  $(d = 23.7 - 58.2 \mu m)$  in comparison to grains within studied calcite veins (d = 450.5-516). The grains-grain size (d) diameter is in positive relationship with twin-length (1), which ranged between 14.27–38.675 µm in marbles and 770.8-972.35 µm in calcite veins. Twin-length (1) and twin-thickness (t) represent key-parameters at calculations of V<sub>twins</sub> – Volume of twins (Rowe & Rutter, 1990).

According to Burkhard (1993), taking into consideration the twin-thickness in marbles (t = 0.8–1  $\mu$ m), we have estimated the low temperature conditions (T< 200 °C), whereas in the case of deformed calcite veins with twin-thickness ranging between 9.43–15.9  $\mu$ m we estimated the higher temperature conditions (T > 250 °C). In calcite veins there were revealed also related manifestations of GBAR – grain boundary area reduction mechanism = related to static recrystallization (Passchier and Trouw, 1996).

Visualizations in the paper show relationships between different parameters. The results are documenting the deformation paleo-conditions, which were "frozen" in studied samples, and such represent a contribution to reconstruction of geodynamic history.

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## Odhad paleonapäťových a paleoteplotných podmienok deformácie zŕn kalcitových agregátov: príklady z Vnútorných Západných Karpát

Deformačné lamely v kalcitových horninách či žilách sú užitočným nástrojom na určenie paleonapäťových a paleoteplotných podmienok zaznamenanej deformácie a rekryštalizácie kalcitových zŕn. Na určenie paleonapätia vyjadreného vo forme diferenciálneho napätia  $(\sigma_D = \sigma_1 - \sigma_3)$  sme použili dve nezávislé paleopiezometrické metódy: Twinning Incidence - metódu početnosti dvojčatenia (I = zdvojčatené zrná/celkový počet zŕn) a Twin Density – metódu hustoty dvojčatenia (D = počet deformačných lamiel/1 mm kolmého priemetu zŕn). Merania boli aplikované na mramoroch príkrovu Bôrky (exhumované relikty meliatsko-hallstattského oceánu) a na kalcitových žilách prestupujúcich celý objem horniny na viacerých odkryvoch v pieninskom bradlovom pásme (sutúrna zóna po penninskom oceáne). Berúc do úvahy tektonický vývoj meliatsko-hallstattskej sutúrnej zóny, paleopiezomtrické výsledky z deformovaných mramorov dokumentujú najvyššie zachované hodnoty diferenciálnych napätí, zatiaľ čo deformované kalcitové žily ako produkt posledných deformačných udalostí poskytujú diferenciálne napätia súvisiace s finálnou deformáciou. V študovaných vzorkách sme realizovali niekoľko sérií meraní, ktoré poukazujú na závislosť kalcitových deformačných lamiel od metamorfných podmienok. Na základe porovnávaní základných parametrov bolo dokázaných niekoľko skutočností.

Priemerná hrúbka deformačných lamiel (t – hrúbka lamiel) rastie s rastúcou teplotou (T). Malá hrúbka v mramoroch (t = 0,8 – 1 µm) je výsledkom nízkoteplotných podmienok kalcitových zŕn (T = 170 – 200 °C) bez ohľadu na napäťové podmienky. V porovnaní s tým kalcitové žily s hrúbkou t = 9,4 – 15,9 µm poukazujú na omnoho vyššie paleoteplotné podmienky (T > 250 °C). Veľkosť kalcitových deformovaných zŕn sa s narastajúcim diferenciálnym napätím ( $\sigma_D$ ) zmenšuje, pričom počet deformačných

lamiel na 1 mm kolmého priemeru zrna (D) sa s narastajúcim diferenciálnym napätím zväčšuje. Malá veľkosť zŕn v mramoroch (d =  $23,7-58,2~\mu m$ ) a veľký počet deformačných lamiel/1 mm (D = 73-646) je dôsledkom deformácie pri veľkom diferenciálnom napätí ( $\sigma_D = 389,37-429,55~MPa$ ). Naopak, väčšie rozmery kalcitových zŕn v kalcitových žilách (d =  $450,5-516~\mu m$ ) a malý počet deformačných lamiel/1 mm (D = 31-52) indikuje nižšie hodnoty diferenciálneho napätia ( $\sigma_D = 203,48-240,44~MPa$ ). Dĺžka deformačných lamiel závisí od veľkosti priemeru kalcitových zŕn (d). Zároveň reprezentuje kľúčový parameter pri výpočtoch  $V_{twins}$  (objemu deformačných lamiel). Nízke hodnoty dĺžky deformačných lamiel (l =  $14,27-38,675~\mu m$ ) v mramoroch sú zapríčinené nízkymi hod-

notami veľkosti zŕn (d = 23,7 – 58,2 µm), čo kontrastuje s kalcitovými žilami, v ktorých bola nameraná veľká dĺžka deformačných lamiel (l = 770,8 – 972,35 µm), narastajúca v závislosti od zväčšujúcich sa rozmerov deformovaných kalcitových zŕn (d = 450,5 – 16 µm). V prípade malej dĺžky lamiel bol vypočítaný objem deformačných lamiel (V twins = 37,7\*10³ – 251,9\*10³) v kalcitových mramoroch omnoho nižší v porovnaní s objemom deformačných lamiel v kalcitových žilách (V twins = 9,9\*106 – 410,7\*106), v ktorých je dĺžka deformačných lamiel omnoho väčšia. Vzájomné porovnania jednotlivých parametrov sú na obr. 3 – 6.

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