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Zircon Recrystallization History as a Function of the U-Content and Its Geochronologic Implications: Empirical Facts on Zircons from Romanian Carpathians and Dobrogea

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1. Introduction

According to Davies et al. (2003), "…the age of Earth and the time scale of pre-human events are central to a civilization's sense of origin and purpose. Therefore, the quest for precise and reliable geochronometers has had a scientific and cultural importance that few other enterprises can match". In this respect, since the beginning of the last century it has been recognized that long-lived radioactive decay systems provide the only valid means of quantifying geologic time.

One of the most reliable Earth's timekeeper has proven to be the mineral zircon, since it records the ages of Earth's earliest evolution stages, the oldest sediments, extinction episodes, mountain-building events and supercontinents' coalescence and dispersal (e.g. Rubatto and Hermann, 2007; Harley et al., 2007). Its widespread use in geochronology is based on the decay of uranium (U) and thorium (Th) to lead (Pb). They provide three distinct radioactive decay series involving the parent isotopes ^{238}U, ^{235}U and ^{232}Th and their daughter isotopes ^{206}Pb, ^{207}Pb and ^{208}Pb, respectively. Through the incorporation of U and Th at the time of growth, every zircon grain hosts three different clocks. In an ideal closed system, the three estimates would agree with each other within the analytical errors of measurements. However, in natural systems the zircon grains are not equally closed for Th and U with respect to post-crystallization effects. The usual approach in zircon geochronology is to consider the U-Pb system alone, as there is no natural non-nuclear ways of fractionating ^{235}U from ^{238}U. As the modern day-ratio of $^{235}U/^{238}U$ is well known (1/137.88) the need to actually analyze very low abundances of ^{235}U is obviated. Besides U and Th, zircon can incorporate some other incompatible elements such as P, Sc, Nb, Hf, Ti, and REE in trace (up to thousands of ppm) or minor (up to 3% wt) amounts. The primary control factor on the substitutions is the ionic radii of the substituting cations compared with Zr^{4+} and Si^{4+} cations. Substitutions that minimize strain effects on either or both sites will be favored. The crystal-chemical limitations are that Zr^{4+} in 8-fold coordination has an ionic radius of $84*10^{-3}$ nm and Si^{4+}, in tetrahedral coordination, has an ionic radius of $26*10^{-3}$ nm. U^{4+} (ionic radius of $10*10^{-2}$ nm in 8-fold coordination) and Th ($105*10^{-3}$ nm in 8-fold coordination) can be accommodated in the Zr^{4+} sites. Uranium concentrations are usually less than 5000 ppm and Th concentration less than 1000 ppm. Because of its ionic radius of $129*10^{-3}$ nm (8-fold coordination), Pb^{2+} is highly incompatible with growing zircon crystal lattice and therefore is not incorporated more than ppb levels, which is crucial in geochronology. Because of the same reason, Pb^{2+} can easily escape from zircon lattice when some conditions are fulfilled.

Based on idea of concordant ages between $^{235}U/^{207}Pb$ and $^{238}U/^{206}Pb$, Wetherill (1956) has developed the Concordia diagram. Quite soon though it has been shown (Tilton et al., 1957) that the concordance situations are rather rare and usually zircon shows evidence of discordance (disagreement between $^{235}U/^{237}Pb$ and $^{238}U/^{206}Pb$ ages) due to Pb loss caused by some post-crystallization geologic events.

Since then, the attempts made for understanding the causes of discordance had become the main preoccupation of the U-Pb geochronologists over the next quarter of century. Finally, it has been understood that discordance can be attributed to two major causes: (1) - mixing, in the analyzed sample volume, of discrete zones of different ages from within the same zircon grain; (2) - partial loss of radiogenic Pb by the entire zircon grain or by fractions of it. While the former cause can be bypassed by in situ dating, the second still hinders unequivocal results.

Continuously growing database of published in situ age data provided further insights into the behavior of U-Pb system in zircons: (1) - radiogenic Pb can be entirely lost and even the concordant data do not indicate the initial crystallization age of zircon; (2) – frequently, Pb loss is accompanied by Th loss; (3) – occasionally, even the U can be lost.

The disturbance of the isotope systematics in zircon is related to: (1) - amorphization; (2) - alteration; (3) - recrystallization.

The focus of the present contribution is on the discussion of the above three processes exemplified with their geochronological consequences on zircons from the Romanian Carpathians and Dobrogea. All data presented here were obtained by in situ dating through Laser Ablation-Inductively Coupled Plasma-Mass Specterometry (LA ICP-MS).

1.1 Amorphization process

This process is due to α-decay events associated with U and Th radioactive disintegration. Based on X-ray diffraction and High Resolution Transmited Electron Microscopy (HRTEM) analysis, Murakami et al. (1991) suggested three stages of damage accumulation in Sri Lankan zircon: in stage I (at $<3 \times 10^{18}$ α-decay events/g) the accumulation of isolated point defects predominate. These defects have the potential to recover through geologic time; stage II (at 3×10^{18} to 8×10^{18} α-decay events/g) is evidenced by crystalline regions with point defects and amorphous tracks caused by overlapped α-recoil nuclei; during stage III (at > 8×10^{18} α-decay events/g) only aperiodic material can be screened by X-ray and electron diffraction.

Salje et al. (1999) suggested a two-phase transition during increasing amorphization process. The first phase is related to the percolation of amorphous material into the crystalline matrix and the second one to the percolation of crystalline material into the amorphous matrix.

According to Nasdala et al. (2001) the α-decay events in the decay chains of U and Th cause the zircon amorphization. An α-particle generates about 120-130 Frenkel type defect pairs along penetration distances from 9.9 to 29.5µm. Recoils of heavy daughter nuclei are only a few hundred Å in length but the recoil damage clusters include 600-1200 Frenkel defect pairs. Spontaneous fission fragments produce heavy damage locally, but their contribution to the overall radiation damage is of minor importance because of their relative rarity. In the absence of recovery, radiation damage is stored in zircon, causing transformation from the crystalline to the metamict state. These authors propose the following stages of radiation damage accumulation.

1. Scattered nano-regions with high defect concentration. The amorphous component is still insignificant.
2. Moderately radiation damaged zircon in which amorphous nano-regions form a domain structure. Amorphous domains in a crystalline zircon or crystalline remnants in an amorphous matrix can be observed.
3. Entirely aperiodic zircon.

Ewing et al. (2003) speak about α-particles ionization processes over a range of 16 to 22 microns that produce several hundreds isolated atomic displacements. The associated α-recoils lose their energy during elastic collisions over 30 to 40 nm, producing localized collision cascades of 1000 to 2000 displacements. However, with increasing temperature the amorphization dose increases. This can be due either, by a decrease in the average cascade size caused by thermal relaxation or by a reduction in the surviving amorphous volume as a result of thermal recovery of irradiation induced defects.

According to Geisler et al. (2007), the amorphization of a crystalline zircon is characterized by transformation from an initial stage, where isolated amorphous domains are surrounded by slightly disordered crystalline material, to a more advanced stage of damage where few isolated, disordered, nano-crystalline islands occur in an amorphous matrix. These authors refer to Salje et al. (1999) when describing the crystalline- to-amorphous transformation as a geometrical phase transition. During the first phase transition, the amorphous domains form clusters that percolate over macroscopic length scales, whereas during the second transition, the crystalline domains cease to be interconnected. The first percolation point appears at an amorphous fraction of ~30% while the second percolation point appears when the amorphous fraction reaches ~ 70%. In other words, less than 30% amorphous fraction and less than 30% crystalline fraction can not be interconnected.

1.2 Alteration processes

According to Geisler et al. (2002), alteration is the interaction between the metamict zircon and fluids, including the meteoric ones (weathering processes), characterized by distinct chemical and structural changes. Geisler et al. (2003) describe two anomalous stages in the alteration rate with increasing degree of amorphization. The first stage takes place when the amorphous domains form interconnected clusters within the zircon structure, namely at the first percolation point suggested by Salje et al. (1999). At this point, the percolation interfaces that represent low-density areas between crystalline and amorphous domains open high diffusivity pathways. A new dramatic increase in alteration rate is observed at the second percolation point of Salje et al. (1999). Around this point, a network of nanometer-size regions of depleted matter interconnects discrete amorphous domains without crystalline dams.

According to Geisler et al. (2007), the structural changes that take place in a zircon grain are defined by the fact that above the 200^o C limit the thermal recrystallization front generated by epitaxial reordering moves inward the zircon crystal. Bellow the 200^o C threshold, the thermal recrystallization involves enhanced defect diffusion only along the reordering front. The above model hypothesizes an increase in the effective diffusion of any species within the zircon lattice with increasing α-decay dose or of amorphization degree.

1.3 Recrystallization

There are several types of recrystallization processes that take place in zircons and several different opinions in terms of its significance exist on this matter.

According to Nasdala et al. (2001), *recrystallization* is a "re-growth" process in the crystallographic sense. During recrystallization a new zircon lattice forms along a crystallization front, typically replacing a more disordered and polluted zircon. Recrystallization leads to healing of the radiation damage and partial or complete resetting of the U-Pb isotopic system. According to the same authors, *annealing* requires only re-formation of disrupted bonds by re-ordering of nearest neighboring atoms, thus annealing of zircon structure is not necessarily associated with any Pb loss. As defined in the literature, *recrystallization* presumes an epitaxial migration of an interface between an ordered region in zircon and a metamict vicinity, therefore it could be described as a defect annihilation or point defect diffusion process. In a general view, Nasdala et al. (2001) enumerate the following thermal recovery mechanisms: (1) - point defect diffusion in the crystalline and amorphous phase; (2) - epitaxial growth of crystalline residuals; (3) - random nucleation in the amorphous phase.

Ewing et al. (2003) put forward a more comprehensive description of the concept of recrystallization, while offering the foundation for a clear distinction between *Type I* and *TypeII* recrystallization. Type I recrystallization is purely thermal and occurs on time scales longer than cascade quench time. This behavior is due to point defect diffusion and epitaxial migration of the crystalline residuals toward the amorphous domains. It can be seen that Type I recrystallization, as described by these authors, covers both the recrystallization and annealing processes of Nasdala et al. (2001). Type I recrystallization prevails over longer periods of time or at higher temperatures and becomes particularly important in natural specimens stored at ambient conditions for geological periods. In moderately damaged zircons, two stages of recovery process have been described. The first stage is defined by the recovery of short-range order and point defect recombination. It occurs bellow ~ 600^o C (Farges, 1994) or ~727^o C (Geisler et al., 2001). The second stage occurs at higher temperatures and is caused by epitaxial recrystallization along the internal crystalline-amorphous boundaries. It is worth noting that an initially moderately damaged zircon grain consists of distorted crystalline phases embedded in an aperiodic matrix at the completion of the first phase. In contrast to the moderately damaged specimens, heavily damaged or amorphous zircon segregates into its constituent oxides at higher temperatures and recrystallization takes place in three stages: (1) - decomposition of amorphous zircon into tetragonal ZrO_2 crystallites and amorphous SiO_2; (2) - tetragonal to monoclinic phase transformation in the ZrO_2 crystallites; (3) - formation of coarse-grained (several hundred µm) randomly oriented, polycrystalline zircon. This process is highly improbable to take place in natural zircon and it excludes the idea of new zircon nucleation within amorphous phase as postulated by Nasdala et al. (2001).

Type II recrystallization occurs as a nearly instantaneous process during irradiation and can be divided into two distinct phases. The first, Type *IIa* recrystallization, is due to increased mobility of interstitials and other point defects during irradiation. The irradiation enhanced diffusion leads to a greater degree of point defect recombination and annihilation. Point defect annihilation is most effective at structural boundaries between amorphous and damaged, but still crystalline regions. Type *IIb* recrystallization occurs within individual displacement cascades. Disordered and highly energetic material can epitaxially recrystallize at the cascade peripheries along with the cooling of the displacement cascade to the ambient temperature. During irradiation, both *Type I* and *Type II* recrystallization processes contribute to the dynamic recovery of zircon, but some mechanisms can prevail over others in certain temperature regimes. Obviously, at the Earth's surface conditions, both Type I and Type II recrystallization processes are less effective than the irradiation damage over an α-dose range, allowing amorphization accumulation in time.

Geisler et al. (2007) proposed two more kinds of zircon recrystallization, fluid or melt assisted. The Hf, U and Th content of zircon can be also distributed in solid solutions between zircon and hafnon ($HfSiO_4$), coffinite ($USiO_4$), thorite ($ThSiO_4$). Because solid-state exsolution structures have not been yet reported in zircon, it is argued that such solid solutions are metastable after cooling and characterized by structural strain. Such structural strain enhances surface reactivity and thus the dissolution rate. We note that the effects of the structural strain are supplementary added to irradiation damage, increasing the reactivity between the zircon and fluids. It was shown (Geisler et al., 2003) that the treatment of radiation-damaged zircon crystals in various aqueous solutions produces inward-penetrating, irregular, and curved reaction domains that resemble those found in natural zircon. Recrystallization of zircon on the expense of the amorphous phase inside the reacted domains occur at experimental temperatures above 200° C. Recrystallization of amorphous zircon dramatically reduces the molar volume of the reacted areas inducing a strain that is partially released by fracturing. Porous structure at nanometer-scale level is also very likely to occur. The nanoporosity created between the crystallites provides pathways for chemical exchange between reaction front and the fluids. This is the diffusion-reaction process in which a moving recrystallization front follows at some distance behind the percolation-controlled, inward diffusion of a hydrous species. According to Geisler et al. (2007), a prerequisite for the diffusion-reaction process is the presence of more than 30 % amorphous fraction, which is of an interconnected amorphous network.

The coupled dissolution-reprecipitation is a process by which the breaking of the bonds and dissolution is accompanied by contemporaneous nucleation and precipitation of new zircon. The coupled dissolution-reprecipitation process is independent of the absolute solubility of zircon in natural aqueous fluids, which is very low (Tromans, 2006). This can result in a complete replacement of one zircon crystal by a new one within the same space, without losing the external shape or crystal morphology (Putnis, 2002; Putnis et al., 2005). The chemical exchange between the dissolution-reprecipitation front and external fluid is maintained by the formation of porosity. This porosity results from the higher solubility and the higher molar volume of the dissolved parent zircon, as compared with the more pure zircon, chemically reprecipitated . Since the dissolution of a metastable zircon solid solution is kinetically favored by structural strain, the radiation damage may also enhance kinetically a coupled dissolution-reprecipitation process. Considering the two proposed mechanisms, the reaction of zircon with fluids and melts provides an effective way of its re-equilibration.

1.4 Temperature conditions of zircon recrystallization

According to Mezger and Krogstad (1997), the 600-650° C temperature interval is a good estimate for recrystallization of damaged zircon. Consequently, lattice damage through α-decay and spontaneous fission may accumulate bellow this temperature. The α-recoil tracks in minerals recrystallize at similar temperature as tracks formed by spontaneous fission fragments (e. g. Murakami et al., 1991). And because fission tracks are retained in zircon up to 200-250° C (Tagami and Shimada, 1996), the α-events damage can also be recovered immediately over 200-250° C (Nasdala et al., 2001). According to Ewing et al. (2003), "temperatures as low as 100-200° C seem to be sufficient to produce measurable recovery over extremely long time scales". Such statements are valid knowing that the quantity of radioactive elements is continuously decreasing in zircon through fission process. The same authors suggest that the critical amorphization temperature for zircon should have an upper limit of 460 K, or 187° C. This temperature is close to the interval indicated by Tagami and Shimada (1996) for recovery initiation. However, Ewing et al. (2003) say that "depending on the mass of the incident ions, the critical amorphization temperature for zircon is between 527 and 750° C". This statement can be understood in the context of external ionic bombardment. Commenting the data of Meldrum et al. (1998), Cherniak and Watson (2003) say that "the critical amorphization temperature for zircons with 1,000 ppm U is about 360° C, and only about 20° C higher for zircons with as much as 10,000 ppm U; and it varies as a function of zircon age by less than a degree per billion years for a given U content. Zircon exposed to temperatures bellow the critical amorphization temperature can accumulate radiation damage, but only over long time scales".

1.5 Isotope systems resetting

According to Mezger and Krogstad (1997) Pb-loss in zircon may occur in four distinct ways:(1)-diffusion in metamict zircon;(2)-diffusion in pristine zircon;(3)-leaching from metamict zircon;(4)-recrystallization of metamict zircon. Pb-loss can be also accompanied by U and Th loss. As mentioned, Geisler et al. (2007) discussing re-equilibration of zircon in aqueous fluids and melts describe two more processes in such environments: (5) - diffusion-reaction process and (6) - coupled dissolution-re-precipitation process. Possibly, leaching from metamict zircon is always accompanied by diffusion-reaction.

According to Cherniak and Watson (2003), cation diffusion in pristine zircon appears to be exceedingly slow under normal crustal conditions. They mention that the closure temperature for Pb in zircon of 100 μm effective diffusion radius for a cooling rate of 10° C/Ma is 991° C. Field based studies showed that Pb-diffusion in the pristine zircon lattice is insignificant around 950-1000° C (e.g. Black et al., 1986; Williams, 1992). Thus, we do not further explore the possibility of isotope systems resetting in pristine zircon.

Regarding the diffusion in metamict zircon, Davies and Paces (1990) and Heaman et al. (1992) observed that even metamict zircon can retain Pb if the temperature is low enough to inhibit recrystallization or there has been no chemical attack of the metamict parts.In conclusion, resetting of U, Th, and Pb istope systems is strongly dependent on leaching and recrystallization of metamict zircon.

Accumulation of radiation damage in zircon is a competition between the α-dose induced disorder (as a function of its content in U and Th) and recrystallization processes. With increasing temperature the irradiation defects become gradually neutralized by instantaneous reordering of lattice, which is equivalent with *Type II* recrystallization. When no new radiation defects accumulate, the critical amorphization temperature is reached. However, we should stress out that most of the previously amorphizated lattice volume is preserved at the critical amorphization temperature. As mentioned before, there is little consensus with respect to the critical amorphization temperature, which is also a function of the α-dose. If the data of Meldrum et al. (1998) are applicable for natural zircon, then the accumulation of amorphization in grains containing 10,000 ppm U is possible bellow 380^{o} C only. In addition to that, if zircon contains 100 ppm U or less, the amorphization becomes improbable (e.g. Mezger and Krogstad, 1997), as a consequence of the low α-dose. Above the critical amorphization temperature and especially above 600-650^{o} C, *Type I* recrystallization will recover the zircon's structure (e.g. Mezger and Krogstad, 1997). During this process, Pb, Th, and U will be variably lost from zircon lattice. If the metamict zircon interacts with fluids, Pb, Th, and U can be lost at temperatures lower than 200^{o} C due to structural recovery front of low temperature *Type I* recrystallization, and over 200^{o} C due to a moving recovery front of high temperature *Type I* recrystallization (Geisler et al., 2007). The diffusion-reaction process causes only partial loss of radiogenic Pb. According to Geisler et al. (2007) an unambiguous chemical indication of the alteration of radiation-damaged zircon by a diffusion-reaction process is the enrichment in Ca, Al, and Fe, and also in common Pb. Enrichment in common Pb can be easily recognized in the recent geochronologic data sets. Briefly, to arrive to a more or less metamict state, which is a function of its U and Th content, and to allow to its isotope systematics to be disturbed, zircon should have: (1) - remained, in geological time terms, bellow the critical amorphization temperature; (2) –reheated subsequently over the critical amorphization temperature; (3) – interacted with aqueous fluids independently of temperature and/or with melts.

2. U, Th, and Pb isotope systems in granites

Dating granites is not always an easy enterprise. They can have a lot of inherited zircons, some of these could have recrystallized in granitic magma and the true magmatic zircons could have been isotopically destabilized at various degrees, depending on their U content and/or on the interaction with fluids. Different aspects of these questions will be exemplified with the Carpathian granites and migmatites.

1. *Variscan granites in the Danubian Domain of Romanian South Carpathians.* Peri-Amazonian basement of the Alpine Danubian Domain of South Carpathians (Balintoni et al., 2011) was massively intruded by granite bodies and their accompanying dyke-swarms (Berza and Seghedi, 1983). There are Cadomian granites with their migmatic escort (Grünenfelder et al., 1983; Liégeois et al., 1996; Balintoni et al., 2011) and Variscan bodies accompanied by cross-cutting dykes (Balica et al., 2007; Balintoni et al., 2011).

From the Variscan intrusions we will further consider the *Buta* pluton (sample 266)* for its low U and isotopically undisturbed zircons in comparison with *Cherbelezu* and *Sfârdinu* plutons defined by high U and strongly isotopically disturbed zircons.

The grains in sample 266 are characterized by: (1) - an U-content in zircon dominantly less than 400 ppm; (2) - a high $^{206}Pb/^{204}Pb$ ratio; (3) - a small U/Th ratio; (4) - nearly concordant ages (Fig. 1).

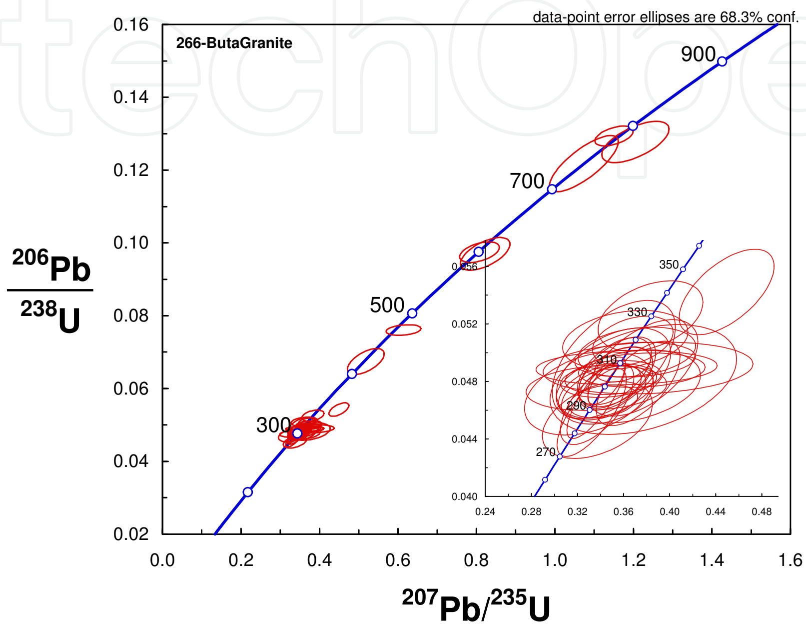


Fig. 1. Concordia projection for sample 266. Good concordances and great concentration around 300 Ma can be noticed. Inset: distribution of ages around 300 Ma.

The vast majority of these zircons represent originally Variscan magmatic grains. Twenty seven $^{206}Pb/^{238}U$ apparent ages ranging between 288.9±7.7 Ma and 314.8±4.3 Ma yielded a crystallization weighted mean age of 303.7±2.4 Ma and a Concordia age of 303.8±0.85 Ma (Fig. 2a, b).

Cherbelezu puton data presented further on were yielded by sample 227. The grains from sample 227 are characterized by two data sets with different parameters: older than 315.2±3.3 Ma and younger than this age. The younger grains with ages ranging between 295.2±3.1 Ma and 122.5±15.1 Ma show: (1) - higher U-content; (2) - higher content in ^{204}Pb; (3) - much greater discordances than the older ones (Table 1 and Fig. 3).

In our interpretation, the older ages suggest inherited recrystallized grains in the Variscan granitic magma. Their isotope systems were completely reset by high temperature *Type I* recrystallization without fluid intervention. The zircons lost all their radiogenic Pb (i.e., good concordance of the data) and partially Th. The available data doesn't allow us to draw some conclusion with respect to any potential U loss. The younger grains most likely represent true Variscan magmatic zircons. Due to their high U content they were amorphizated post 300 Ma and partially lost radiogenic Pb (i.e., poor concordance of the data) and Th during the Alpine thermotectonic events, while gaining ^{204}Pb. The isotope

* Analytical data are available upon request from the authors

systems were probably disturbed in the presence of fluids and amorphization grade reached the first percolation point of Salje et al. (1999). The data suggest that a diffusion-reaction process was active due to structural recovery front of low temperature *Type I* recrystallization, bellow 200º C. The true zircons' crystallization time was probably less than 315.2±3.3 Ma and more than 295.2±3.1 Ma. Thus we suggest for Cherbelezu pluton a crystallization age closed to that of Buta pluton.

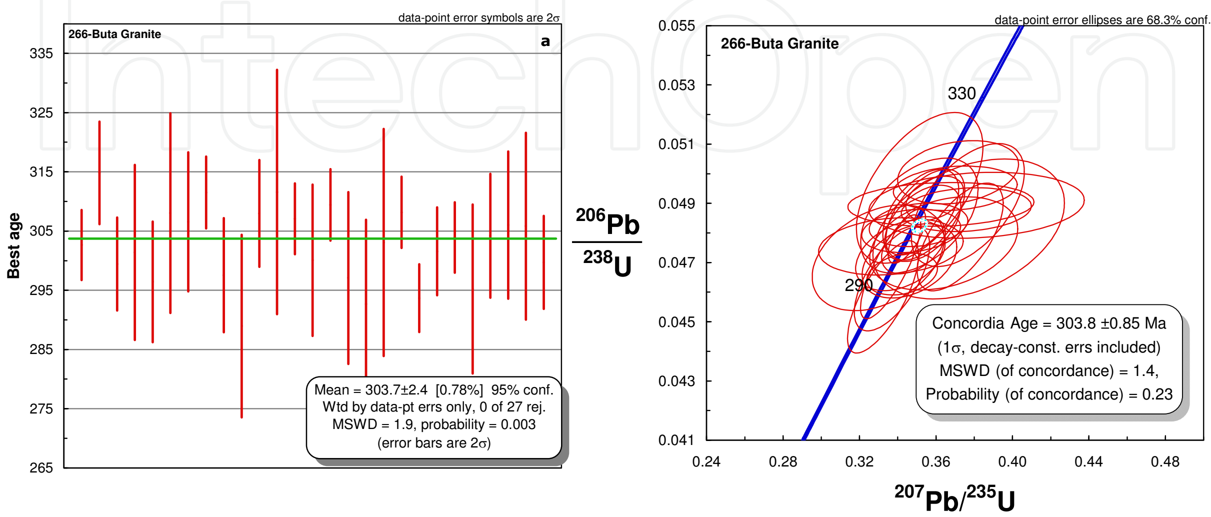


Fig. 2a, b. Weighted mean age and Concordia age for sample 266.

	Older grains	Younger grains
U average content (ppm)	3990	13623
204Pb average content (ppm)	3.46	87.9
U/Th average ratio	8.5	6.3

Table 1. Comparative isotope parameters between the two sets of grains in sample 227.

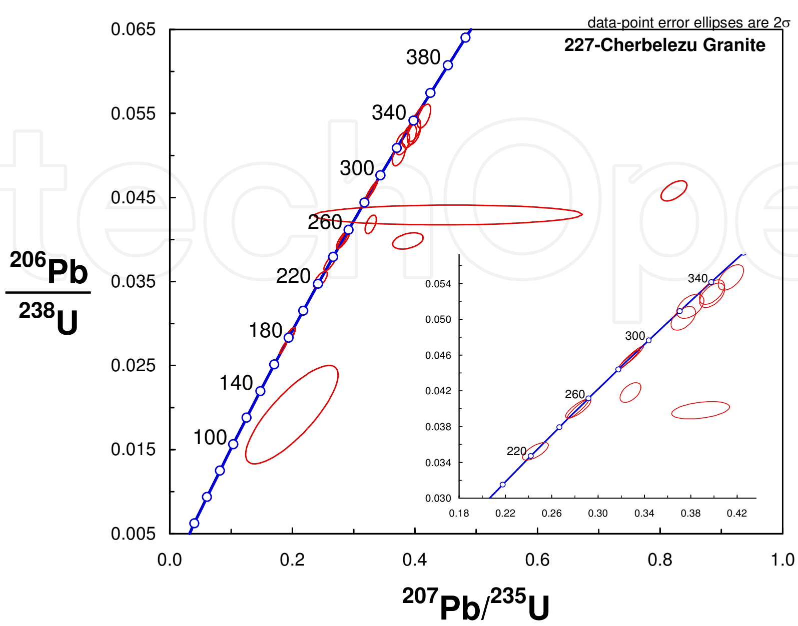


Fig. 3. Concordia projection for sample 227.

The data presented for *Sfârdinu* pluton were provided by sample 229. The grains pertaining to this sample are characterized by: (1) - high U-content (4413 ppm average); (2) - high ^{204}Pb-content; (3) - great discordances for most of the ages, except for 2 out of 50 (Fig. 4); (4) - variable U/Th ratios, generally over the normal ratios in undisturbed magmatic zircon.

Considering the above observations, we infer that all the analyzed crystals represent inherited zircons disturbed initially during the Variscan orogeny, followed by the Alpine thermotectonic events. In their present state all the grains show isotopic disturbance assisted by fluids. Amorphization grade reached the first percolation point of Salje et al. (1999) and grains probably remained bellow 200° C along their entire post Variscan history. Clearly, also in this case, the grains with more than 5000 ppm U gained much more ^{204}Pb than the grains with less than 5000 ppm U. The crystallization age of Sfârdinu pluton is difficult to ascertain. If we consider the most concordant age sets (between 305.4 and 318.6 and between 292.1 and 305.6 Ma) we get a weighted mean age of 301.5±6 Ma (Fig.5).

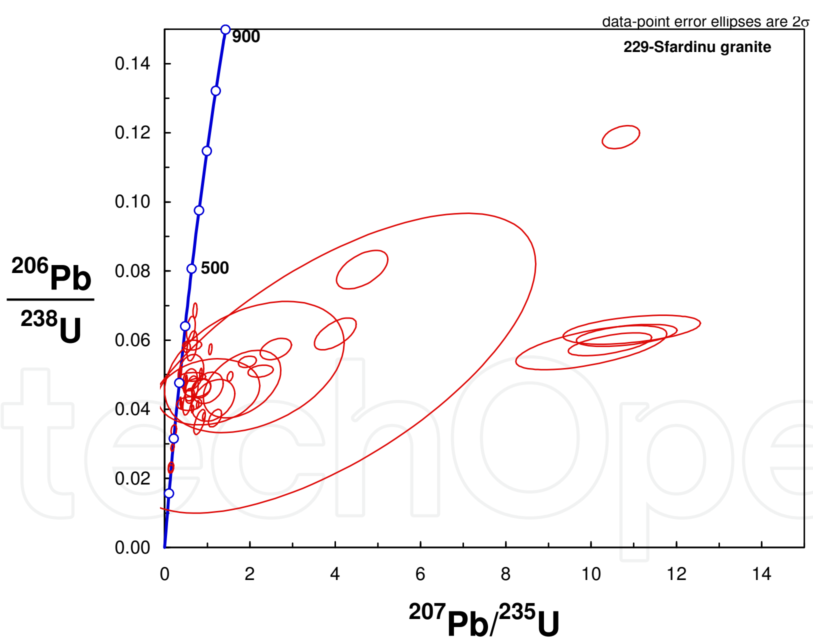


Fig. 4. Concordia projection for sample 229.

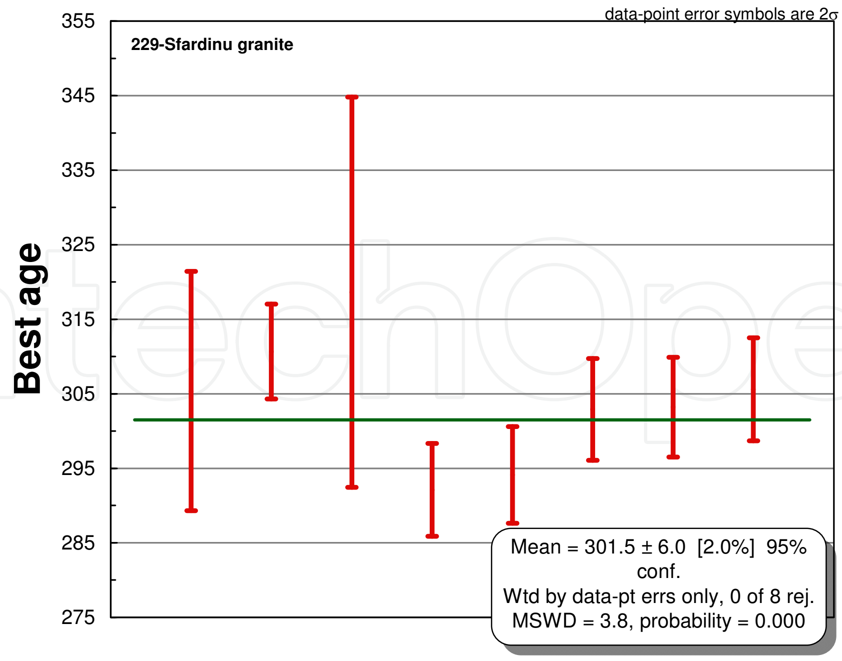
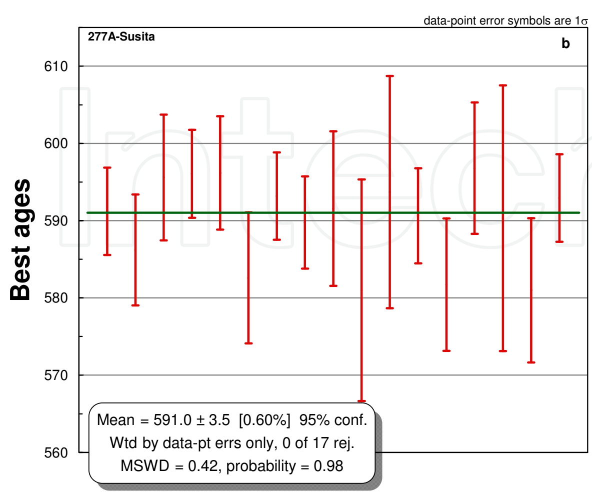


Fig. 5. Weighted mean age for sample 229.

2. *Cadomian granites and migmatites.* Grünenfelder et al. (1983), Liégeois et al. (1996), and Balintoni et al. (2011) dated the Tismana, Şuşiţa, Novaci, and Olteţ plutons and their results bracketed the ages around 600 Ma. These plutons cross-cut a dense swarm of migmatic dykes characterized by black K-feldspar grains (Berza and Seghedi, 1983). Out of the four Cadomian plutons we exemplify the relationship between U-content and isotope disturbance in Şuşiţa pluton. Two samples (277A and 277B) collected several km apart each other show quite different isotope data.

Using the data from sample 227A, Balintoni et al. (2011) obtained a crystallization weighted mean age of 591.0±3.5 Ma and a Concordia age of 591.6±1.8 Ma (Fig. 6a, b).



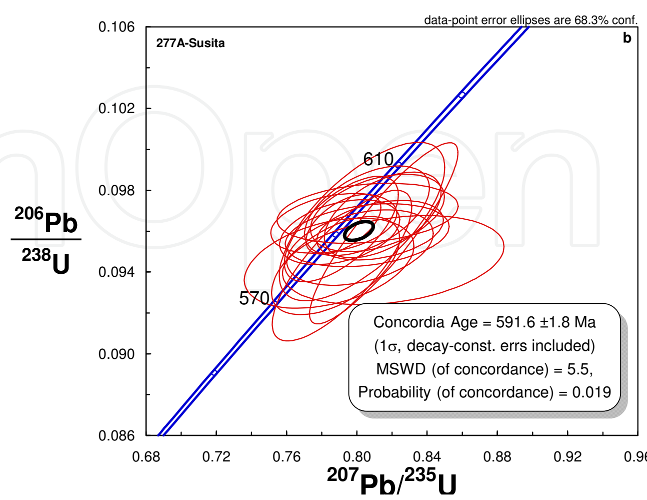


Fig. 6a, b. Weighted mean age and Concordia age for sample 277A.

In comparison with the grains from sample 277A, the grains from sample 277B show: (1) - higher U-content; (2) - higher U/Th ratio; (3) - lower $^{206}Pb/^{204}Pb$ ratio; (4) - greater discordances (Fig.7); (5) - complete lack of protolith ages. Judging from the age discordances, is safe to assume that all the ages were partially reset. The comparative parameters are presented in Table 2.

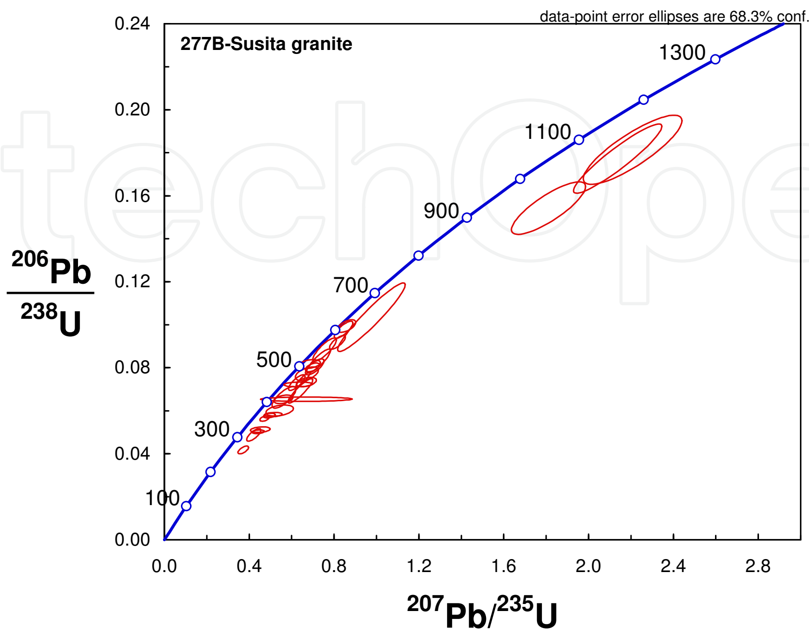


Fig. 7. Spread of ages along Concordia for sample 277B. No ages around 600 Ma.

	Sample 277A	Sample 277B
U average content (ppm)	392	1412
U/Th average ratio	3.2	22.7
$^{206}Pb/^{204}Pb$ average ratio	71374	42429

Table 2. Comparative isotope parameters between the grains in samples 277A and 277B.

Zircons from sample 277B lost radiogenic Pb and Th, and gained ^{204}Pb. There is a direct corellation between U-content, ^{204}Pb gain, age rejuvenation, and concordance deterioration. Therefore, for ages bellow 400 Ma the U average content is of 1883 ppm and $^{206}Pb/^{204}Pb$ average ratio is 27351. For ages between 400 and 500 Ma, the average content of U is of 1235 ppm and the $^{206}Pb/^{204}Pb$ average ratio is 36560. For sample 277B we infer an amorphization grade of up to 40 % and *Type I* recrystallization by point defect diffusion under moderate influence of fluids, as the main recrystallization mechanism. According to Geisler et al. (2007), we assume a diffusion-reaction process at temperatures bellow 200^{o} C. The inferences made for the sample 277B are strongly sustained by the data from the samples 334 (Fig. 8) and 10-406 (Fig. 9) representing Cadomian migmatic material.

The main conclusion that can be drawn is that by increasing the U-content, the isotopic parameters become gradually deteriorated (e.g., discordances in Figs. 8 and 9), while making a distinction between the Variscan and Alpine reworking virtually impossible. At higher U contents the radiogenic Pb and Th are lost in greater quantities and concomitantly more common Pb is added as portrayed in Table 3.

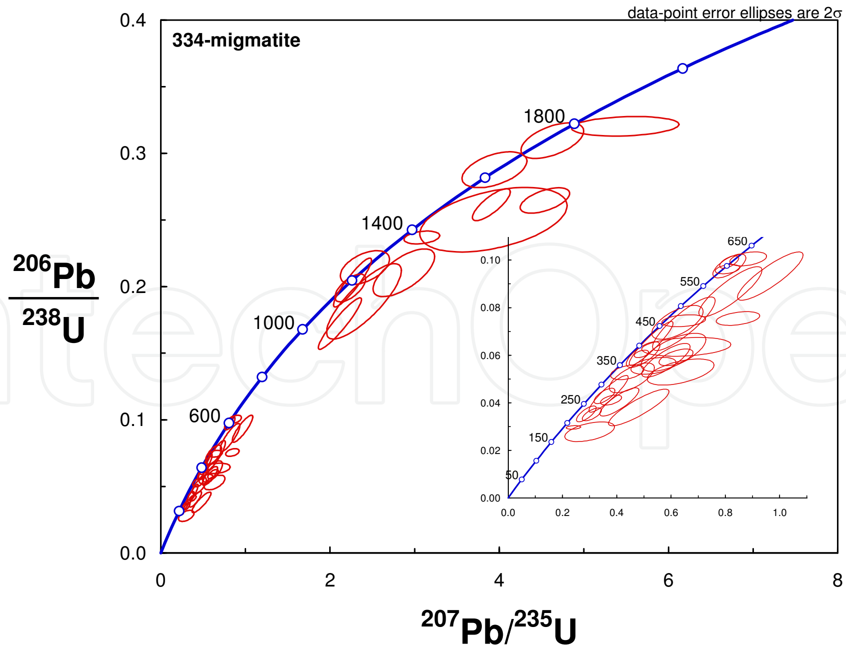


Fig. 8. Spread of ages along Concordia for sample 334. All the magamatic ages have been reset.

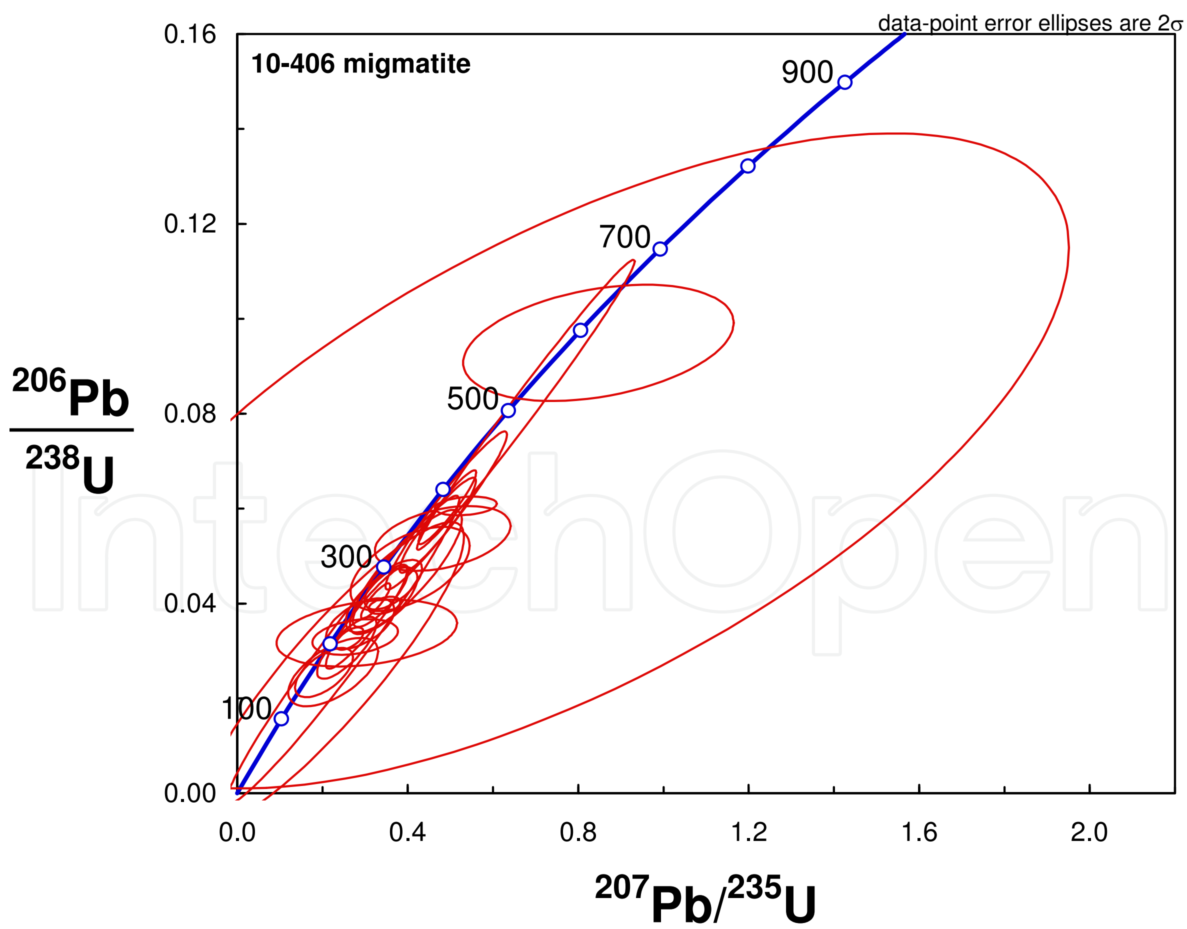


Fig. 9. Spread of ages along Concordia for sample 10-406.Again all the magmatic ages have been reset.

Sample 334		
Ages (Ma)	U-average content (ppm)	U/Th average ratio
>617	385	2.6
617-383	1236	23.9
<383	2049	55.5
Sample 10-406	4465	115.7

Table 3. Comparative isotope parameters between sets of grains from sample 334 and the same parameters for the grains from sample 10-406.

No age was completely reset and the involvement of fluids in these processes is highly suspected. We postulate a poor zircon lattice recovery by defect diffusion processes but the amorphizated material did not recrystallized. During post Cadomian events the grains remained bellow the amorphization critical temperature, very likely bellow 200^{o} C. In the case of sample 10-406 the massive loss of Th and gain of common Pb can be interpreted as an amorphization grade above the second percolation point of Salje et al. (1999), that is more than 70 % amorphization.

3. U, Th, and Pb isotope systems in orthogneisses, metamorphosed under medium grade conditions

The igneous protoliths of orthogneisses are isotopically stabilized during metamorphism. However, as a function of the U-content, temperature history, and fluids intervention, the isotope systems can potentially be reset. Several examples from Apuseni Mountains and East Carpathians will illustrate the behavior of zircon in orthogneisses during Ordovician initial metamorphism and during later Variscan thermotectonic events.

1. *Orthogneisses from the basement of Someş pre-Alpine terrane Apuseni Mountains.* The geochronology of the Apuseni Mountains pre-Alpine terranes has been detailed by Balintoni et al. (2010b). The Someş terrane basement of Ordovician age was intruded by Variscan granites when it functioned as an upper plate. We will further discuss the data from the samples 166 and 167, representing the same orthogneiss body.

The grains from the sample 166 show good concordances (Fig.10), a low ^{204}Pb, small U/Th ratios, and variable U-content. Only three grains show strong reset of U/Pb isotopic system during the Variscan thermotectonic events, with an U average-content of 1642 ppm. The majority of zircon grains preserve Ordovician ages, yielding a weighted mean age of 452.3±5.2 Ma and a Concordia age of 452.4± 3.7 Ma (Fig. 11a, b). The data from sample 166 suggest a Type I recrystallization process of higher temperature of the amorphizated grains, without fluids intervention.

At an U average-content of 2002 ppm, all the grains from the sample 167 lost the radiogenic Pb while the youngest of them gained ^{204}Pb. Rejuvenation lead to the deterioration of the concordances (Fig. 12) but the U/Th ratio remained relatively constant. This is further proof that amorphization grade and resetting of the isotopic systems are a function of the U-content. In the case of sample 167, we advocate for a *Type I* recrystallization at a temperature over the critical amorphization threshold.

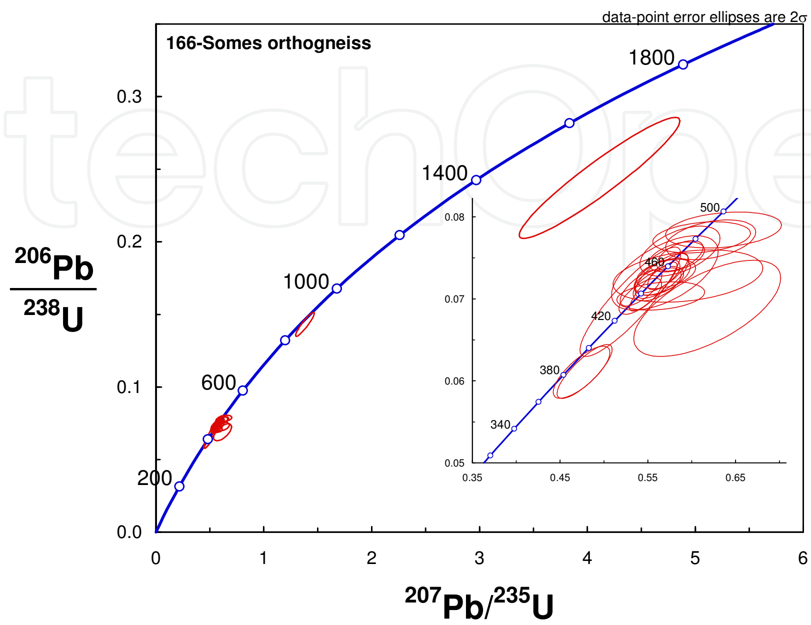


Fig. 10. Concordia projection for sample 166.Concordant ages around 450 Ma.

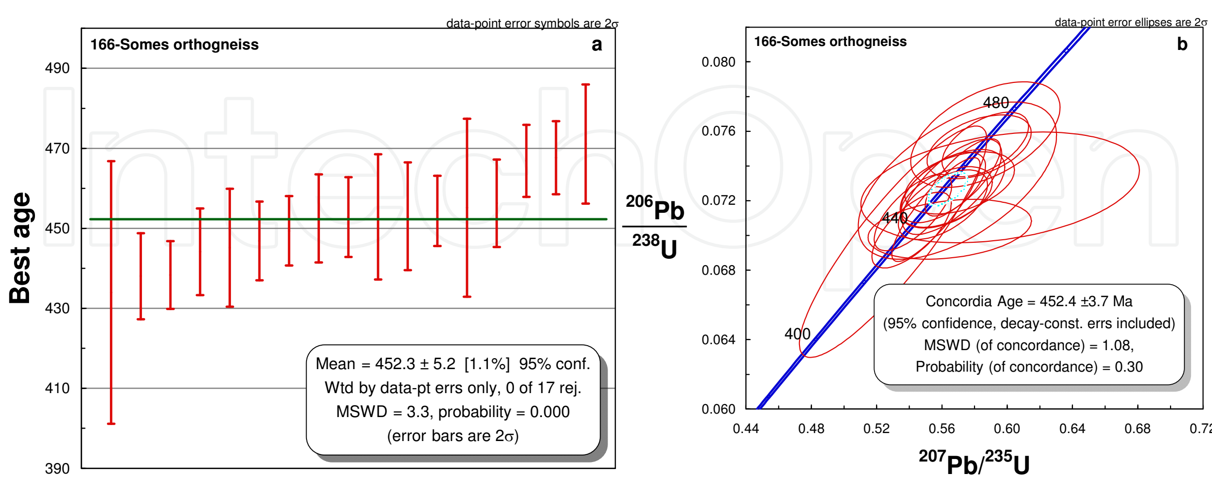


Fig. 11a, b. Weighted mean age and Concordia age for sample 166.

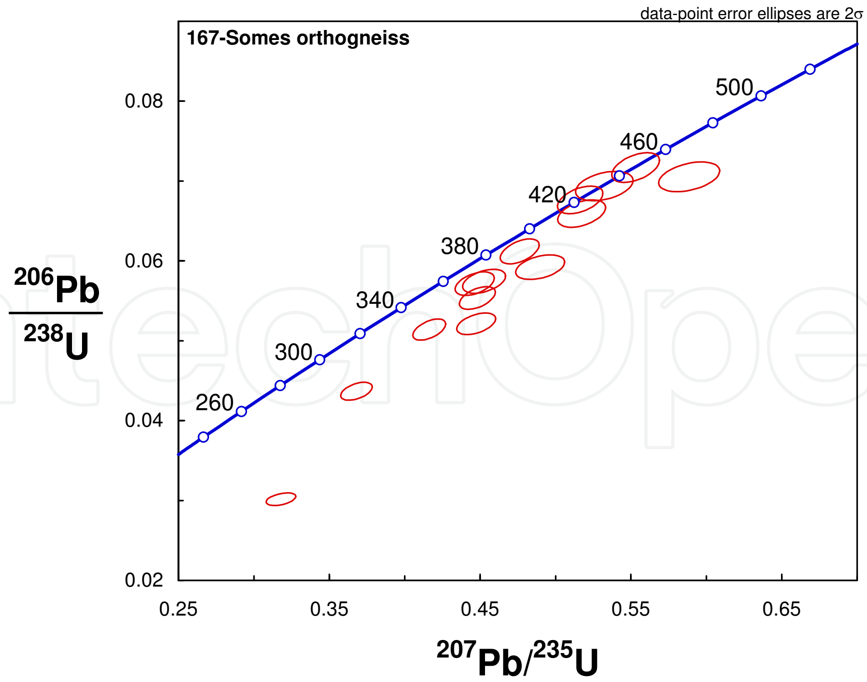


Fig. 12. Spread of ages along Concordia for sample 167.Reset and discordant ages at higher U-content.

2. *Orthogneisses from the basement of the East Carpathians pre-Alpine terranes.* Preliminary geochronologic data on the basement of the pre-Alpine terranes from East Carpathians were presented by Balintoni et al. (2009). In this contribution we will focus mainly on the data from the basement of the Tulgheş terrane and from the basement of the Rebra terrane represented by Negrişoara metramorphic unit. These terranes composed the median part of the Variscan nappe pile and were affected by retrogression down to the chlorite grade temperature (Balintoni, 1997) when the K/Ar ages were reset around 300 Ma (Kräutner et al., 1976).

Tulgheş orthogneiss, sample 10-476. Except a single anomalous age, the data in sample 10-476 show: (1) - good concordances (Fig 13); (2) - low U/Th ratio; (3) - high $^{206}Pb/^{204}Pb$ ratio; (4) - low U-content; (5) - no age younger than the Ordovician. Considering all the Ordovician ages, these can be divided in two distinct groups: (i) between 449.6 and 469.0 Ma; (ii) between 474.4 and 488.8Ma. The younger set corresponds to an U average - content of 203 ppm and to an U/Th average ratio of 3.7., while the older set corresponds to an U average-content of 185 ppm and to an U/Th average ratio of 3.0. The above data suggest a slight increase in the U-content and a decrease in the Th content toward younger ages. Because there is little evidence for any disturbance in the isotopic systems we interpret the age range as an evolution of the magmatic system from its source to the crystallization time. The two data sets yielded a younger $^{206}Pb/^{238}U$ weighted mean age of 462.6±3.1 Ma and an older one of 478.3±5.5 Ma (Fig. 14a, b). We consider the first age to be a better candidate for the protolith crystallization age. There is no doubt that the dated grains constituted isotopically closed systems post protolith crystallization. Therefore, we conclude that bellow the concentration of 250 ppm U, the zircon lattice is prone to recovering even at low temperatures, by *Type* II recrystallization, while the radiation damage and the amorphization can not accumulate.

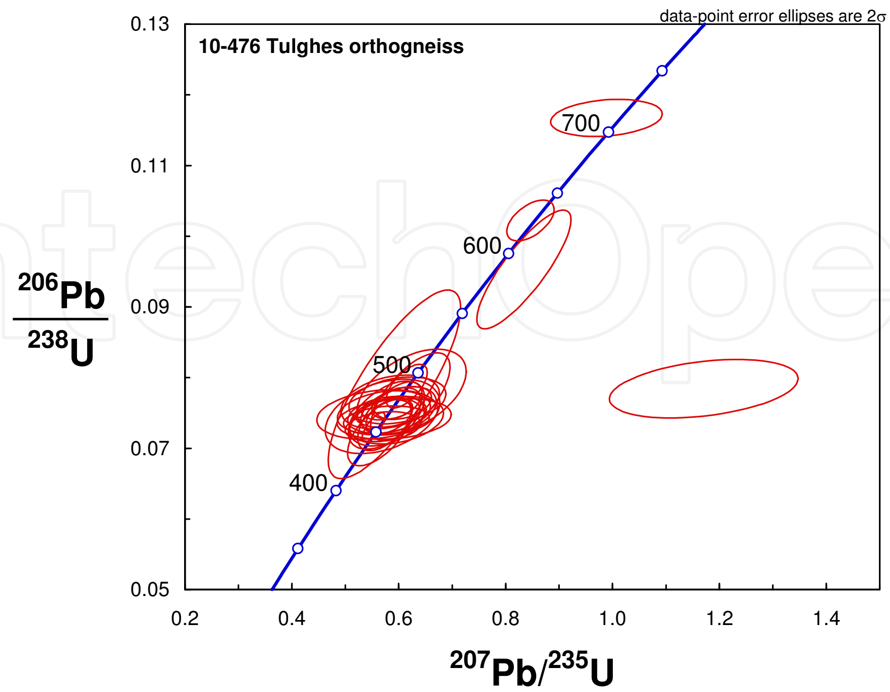


Fig. 13. Concordia projection for sample 10-476.No one age was reset.

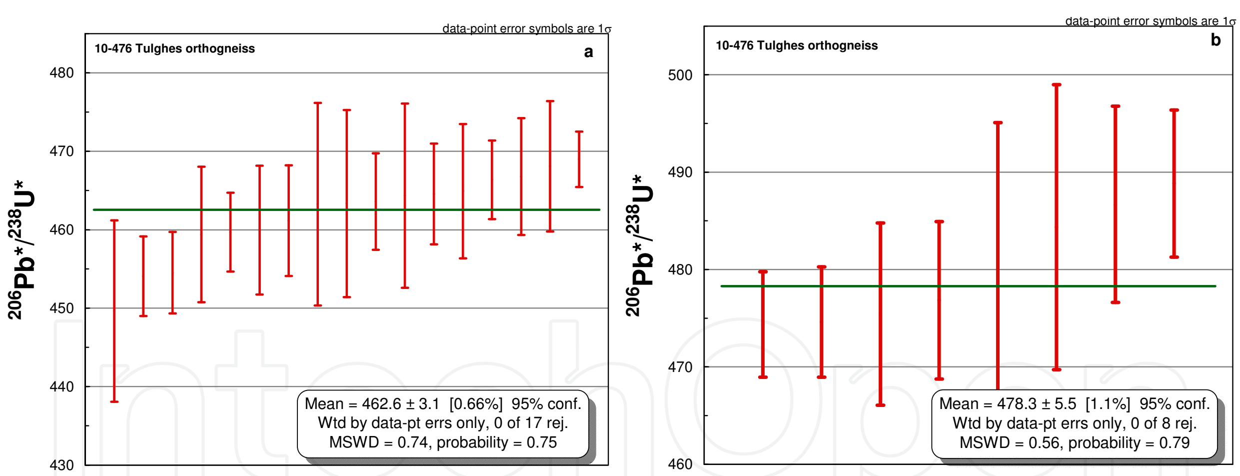


Fig. 14a, b. Weighted mean ages for the two sets of ages from sample 10-476.

Pietrosu Bistriței orthogneiss, Negrişoara metamorphic unit, sample 10-475.

The data from this sample are similar to data from sample 10-476.The younger grains show 226 ppm U in average and an U/Th average ratio of 8.4, while the older grains have an average concentration of 226 ppm U and an U/Th average ratio of 5.5. The younger data set yielded a $^{206}Pb/^{238}U$ weighted mean age of 461.5±5.2 Ma and the older one yielded 477.8±4.2 Ma (Fig. 15a, b). We interpret these ages identically to ages from sample 10-476. As in the case of the previous sample, there is no evidence for zircon damage due to radiation at an U-content bellow 300 ppm.

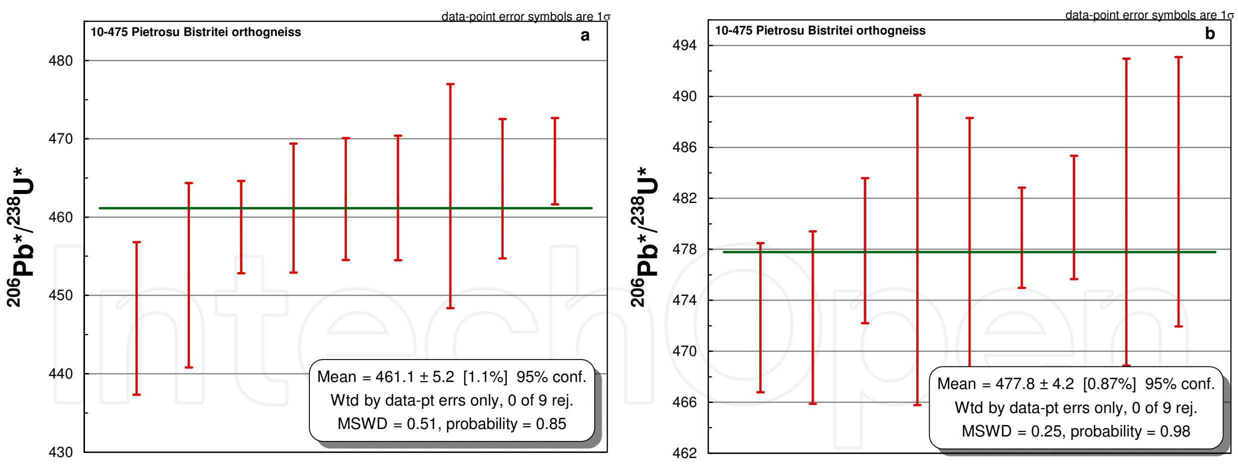


Fig. 15a, b. Weighted mean ages for the two sets of ages from sample 10-475.

4. U, Th and Pb isotope systems in orthogneiss zircons affected by Variscan eclogite-facies metamorphism

Medaris et al. (2003) argued for a Variscan age of the eclogite-grade metamorphism known from the basement of the Sebeş-Lotru pre-Alpine terrane (Iancu et al., 1998; Săbău and Massonne, 2003). Balintoni et al. (2010c) published geochronologic data that revealed the composite nature of the Sebeş-Lotru terrane basement, which consists of Cadomian and Ordovician (Caledonian) igneous protoliths. In the following paragraphs the Cadomian *Frumosu* orthogneiss and *Tău* Ordovician orthogneiss will be discussed.

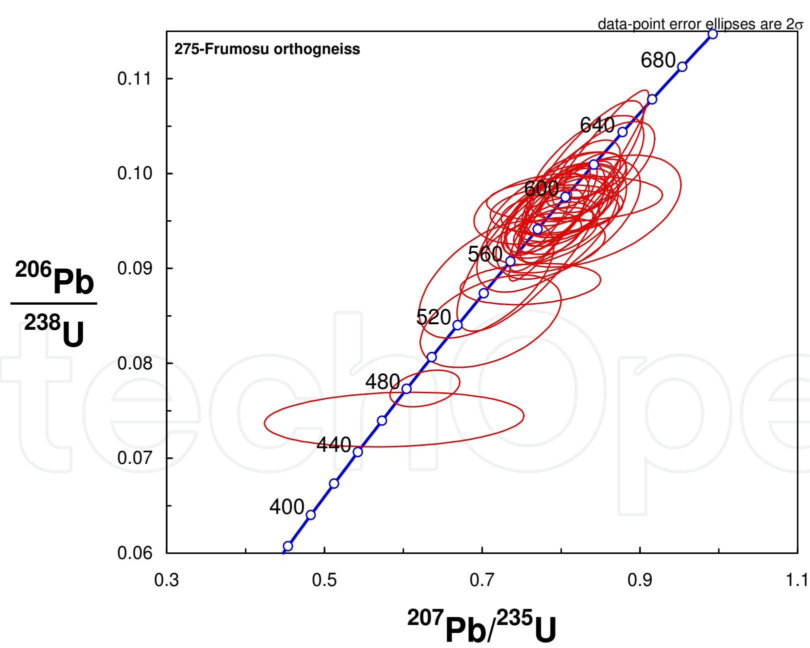


Fig. 16. Concordia projection for sample 275

Frumosu orthogneiss .The grains from sample 275 are characterized by: (1) - very good concordances (Fig 16); (2) - low U/Th ratios; (3) - high $^{206}Pb/^{204}Pb$ ratios; (4) - low U-contents. The U average-content for all grains is around 300 ppm. From 36 dated zircon grains, only 5 of them show Pb loss and 3 of them exhibit Th loss in various degrees. The ages tend to cluster on

Concordia diagram and can be divided in two sets as in East Carpathians orthogneisses, between 568 and 593 Ma and between 597 and 618 Ma. The first data set yielded a $^{206}Pb/^{238}U$ weighted mean age of 584.8±3.6 Ma and the second set yielded a mean age of 606.6±4.4 Ma (Fig. 17a, b). We consider the younger age closer to the crystallization time of igneous protolith while the slightly older mean age is interpreted as an early crystallization event during melt genesis. The main conclusion is that even under the eclogite metamorphic facies conditions, the zircons with less than 300 ppm U do not show isotope systems resetting.

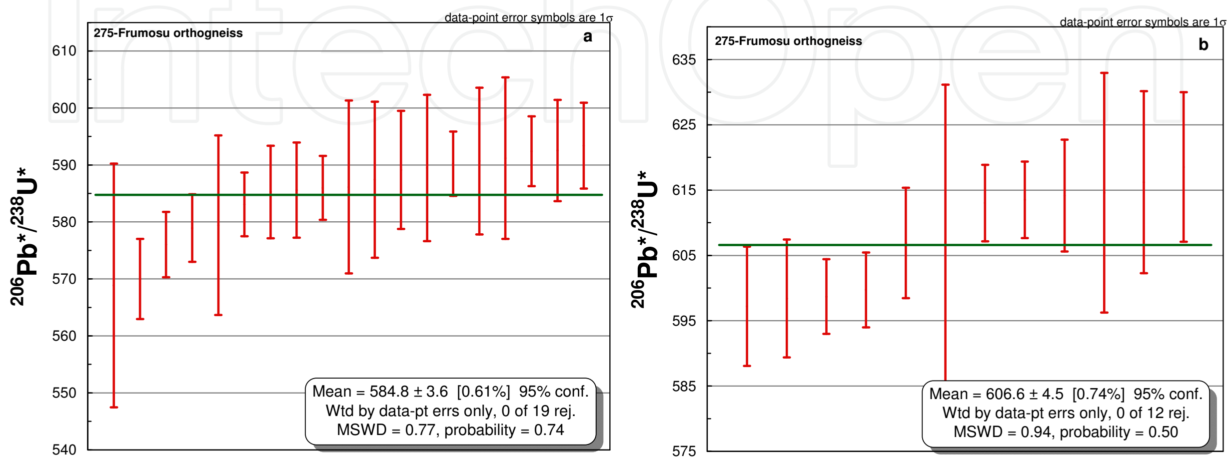


Fig. 17a, b. Weighted mean ages for the two sets of ages from sample 275.

Tău orthogneiss crops out in the median part of the Sebeş valley as a component of the Cumpăna Ordovician metamorfic unit.

The grains from sample 272 are characterized by: (1) - over 1000 ppm U in all the grains; (2) - a deterioration of concordances, yet acceptable for many grains (Fig.18); (3) - a single grain from 36 preserving a protolith age; (4) - Th loss toward the younger ages; (5) - ^{204}Pb gain is in variable quantities and not recorded by all grains.

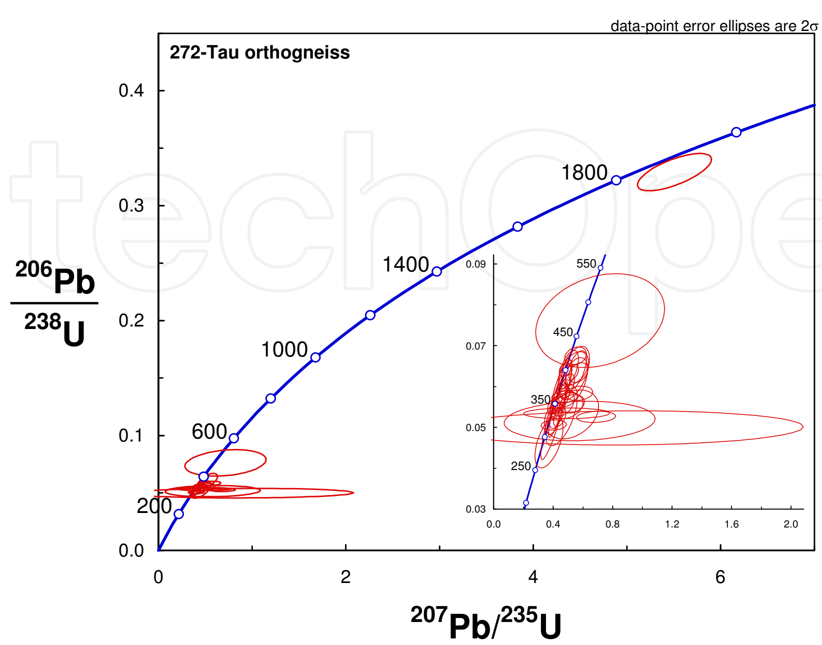


Fig. 18. Concordia projection for sample 272.

It is only obvious that at such high U-content all the grains suffered amorphization prior to the Variscan eclogite event and that during this event they underwent the *Type I* high temperature recrystallization process. The incomplete radiogenic Pb loss can be explained by fluid intervention proved by ^{204}Pb gain.

5. U, Th and Pb isotope systems in detrital zircons from medium grade metaquartzites and paragneisses

Most frequently the detrital zircons remain stable under crustal thermodinamic conditions because they were well selected with respect to their U-content during weathering, transport, and sedimentation. This observation is generally valid for zircons from quartzitic rocks that underwent long and possible repeated sedimentary cycles. In paragneisses, however, the material is often poorly sorted and can originate from proximal sources. The above situations will be exemplified by samples from the pre-Alpine Orliga terrane in North Dobrogea, involved in a Variscan suture as a lower plate (Balintoni et al., 2010a). The basement of the Orliga terrane has been intensely migmatized during Variscan orogenic event, fact that suggests minimum temperatures around 650-700º C. We will begin by scrutinizing the detrital zircons from the sample 336, a metaquartzite.

In this particular case, the U-content in grains is quite low (out of 72 measured grains, 52 have less than 200 ppm U). Furthermore, the U/Th ratio is typical for magmatic zircons (generally smaller than 3.5), the ^{206}Pb/^{204}Pb ratio indicates low ^{204}Pb content, and the concordances are surprisingly good even for early Proterozoic or Archean ages (Fig. 19). These observations confirm the lack of the isotopic disturbances in all the grains. The age data are interpreted to represent original crystallization ages in the zircon sources with no signs of the Variscan thermotectonic events recorded by zircons. The metasediment deposition age is not older than the late Cambrian.

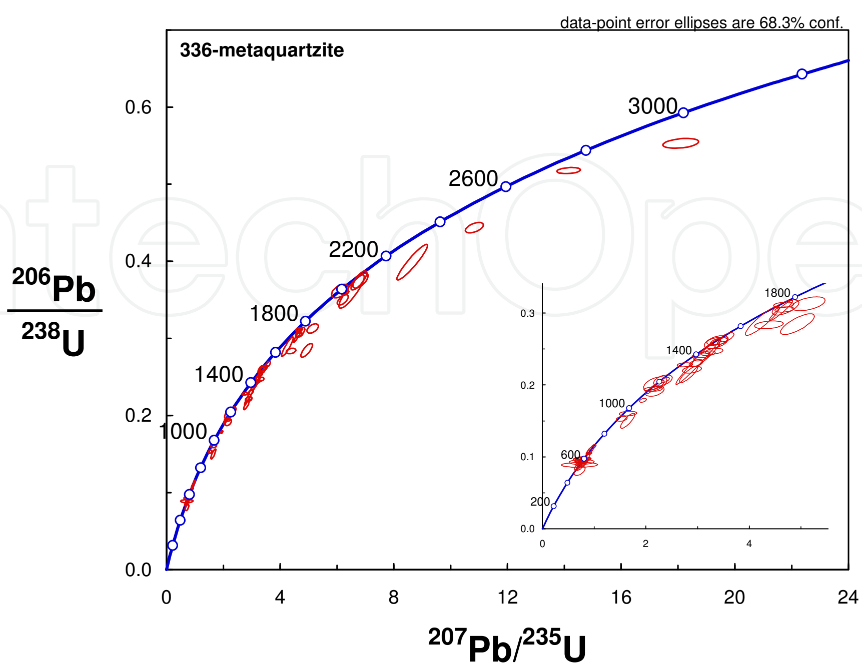


Fig. 19. Concordia projection for sample 336

The zircon grains from sample 167GPS (a paragneiss) are characterized by the followings: (1) - numerous ages clustered around 300 Ma; (2) - variable U-content in zircon grains; (3) - strongly modified U/Th ratio in comparison with the sample 336; (4) - high $^{206}Pb/^{204}Pb$ in all the grains; (5) - good concordances (Fig.20).

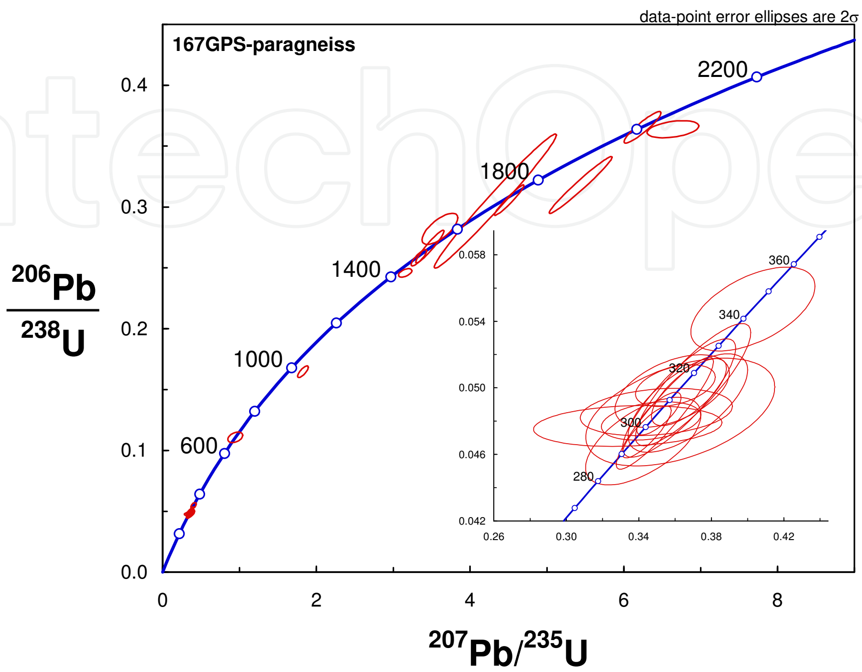


Fig. 20. Concordia projection for sample 167GPS.

The isotopic parameters for the young as well as for the old grains are presented in Table 4.

	Ages between 343.5 -294.8 Ma	Ages older than 600 Ma
U average content (ppm)	686.7	209.9
U/Th average ratio	25.8	2.4

Table 4. Comparative isotopic parameters for different sets of grains from sample 167GPS.

Considering all the data, several conclusions can be drawn. From 26 measured grains, 15 grains recrystallized during Variscan incipient melting. All these grains have an U content usually exceeding 300 ppm U. They lost all the previous radiogenic Pb, a great part of Th, and possible some U. In the same time, the recrystallized grains did not gain any ^{204}Pb and their concordances are remarkable good. These observations suggest a *Type I* recrystallization process by epitaxial migration of the interfaces between the crystalline and amorphizated parts of the grains. The grain lattices have recovered completely without fluid intervention and in presence of a melt. Apparently, ca. 300 ppm U was the boundary between the damaged and undamaged zircons.

A more complex situation is depicted by the data of sample 335.

In great lines the processes are similar to those in sample 167GPS. However, two parameters are more deteriorated in the zircons of sample 335 than in sample 167GPS: many grains gained ^{204}Pb and the ages moved away of Concordia (Fig. 21).

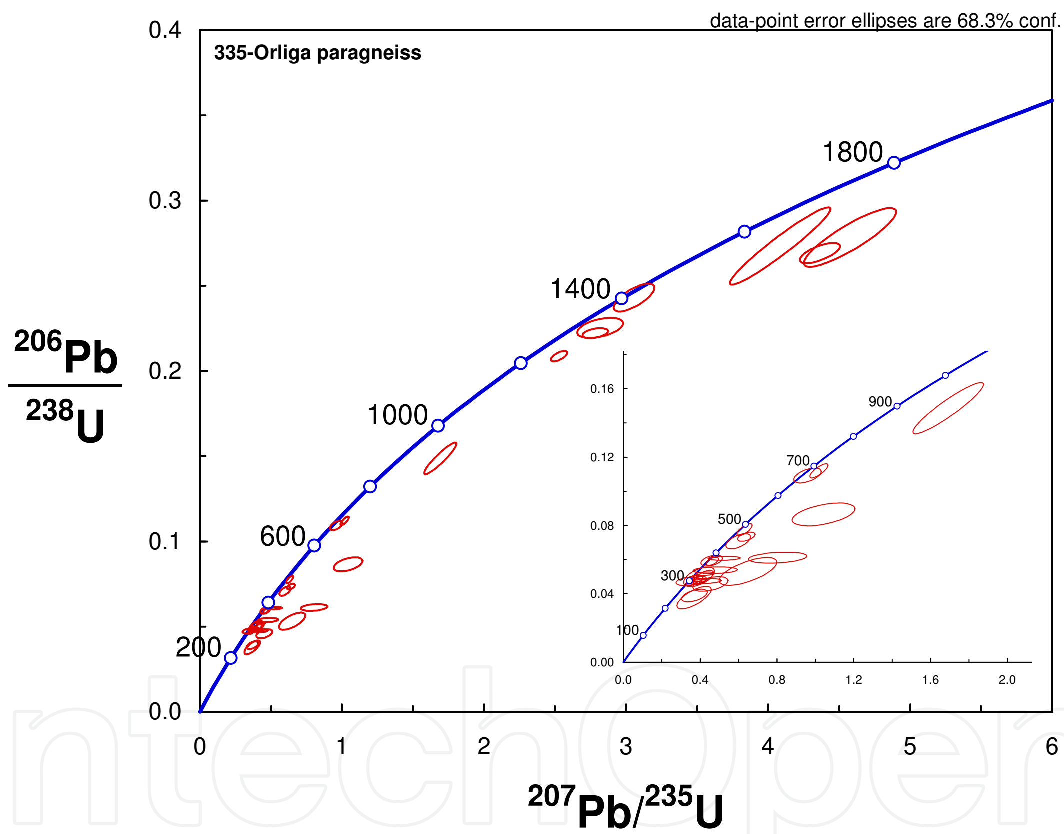


Fig. 21. Concordia projection for sample 335.

These two facts suggest that fluids were extensively involved in the recrystallization processes. Several zircon grains exhibiting ages bellow 300 Ma indicate also later disturbing events.

To see again the role of the U content in zircons history we exemplify by the sample 168GPS.

With the exception of 3 grains (out of 34 analyzed grains), all the other have their U content less than 200 ppm. None of the analyzed grain was isotopically disturbed, clear evidence that at such low U content the effects of the amorphization process are indiscernible and the lattice damage do not accumulate even in geological time. All the grains show the original ages and no sign of the Variscan thermotectonic event is evident (Fig.22).

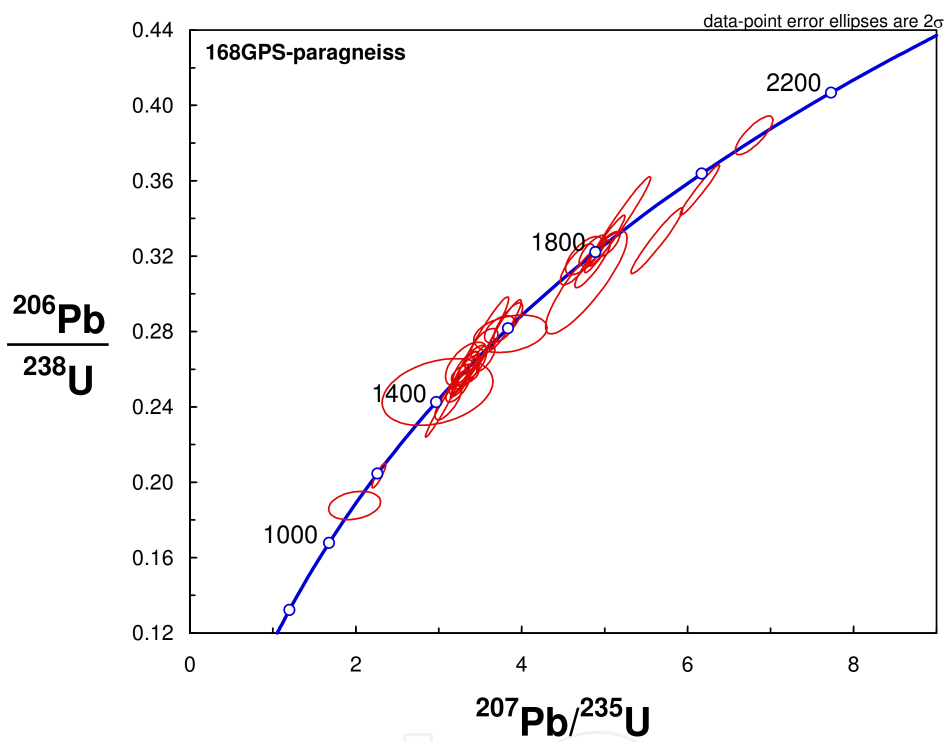


Fig. 22. Concordia projection for sample 168GPS.

6. Conclusions

According to the presented data, the boundary between accumulation of lattice damage and continuous recovery, below the critical amorphization temperature can be set at around 300 ppm U concentration. The undamaged lattices with less than 300 ppm U, sometimes show along the Concordia a spread of ages of ca. 40 Ma from which two valid weighted mean of Concordia ages can be obtained. Usually, the grains display a slight increase in the U-content and U/Th ratio toward the younger ages. The younger age is probably closer to the real crystallization age of the rock.

The migration of ages with respect to the Concordia, when the zircon grains contain over 300 ppm U, can not be explained easily in many situations. This is at least partly because some of the grains appear to lose all the radiogenic Pb at intermediate stages between well known major thermotectonic events. However, the high U-content grains can be useful in deciphering the thermotectonic history of the rocks whenever they reset dominantly around younger ages. If all the grains from a sample contain more than 300 ppm U, the initial age can be totally reset by the subsequent thermotectonic events.

Both, the magmatic and detrital zircons with over 300 ppm U record the metamorphic events, yet the low-U detrital zircon from metaquartzitic rocks do not reset throughout the crustal thermotectonic history.

Alteration in the presence of fluids strongly promotes the resetting of isotope systems as well as recrystallization even at temperatures bellow the crytical amorphization threshold. The amorphization grade is proportional with the U content, while there is little evidence for the role played by the pressure during recrystallization.

Most frequently, Th is lost together with radiogenic Pb, but U loss is by far less a common phenomenon.

7. Acknowledgements

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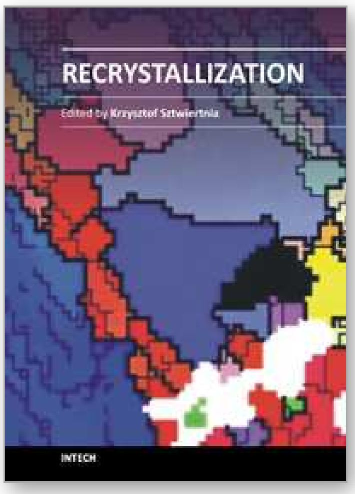

Recrystallization

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Recrystallization shows selected results obtained during the last few years by scientists who work on recrystallization-related issues. These scientists offer their knowledge from the perspective of a range of scientific disciplines, such as geology and metallurgy. The authors emphasize that the progress in this particular field of science is possible today thanks to the coordinated action of many research groups that work in materials science, chemistry, physics, geology, and other sciences. Thus, it is possible to perform a comprehensive analysis of the scientific problem. The analysis starts from the selection of appropriate techniques and methods of characterization. It is then combined with the development of new tools in diagnostics, and it ends with modeling of phenomena.