# APPLICATION OF THE $^{40}Ar/^{39}Ar$ METHODOLOGY IN THE STUDY OF TECTONIC REACTIVATIONS OF SHEAR ZONES: ROMERAL FAULT SYSTEM IN THE CENTRAL CORDILLERA OF COLOMBIA

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## INTRODUCTION

The $^{40}Ar/^{39}Ar$ methodology is an applicable tool to a wide spectrum of geological problems (McDougall & Harrison, 1999; Renne, 2000). In the case of thermocronology of shear zones (Goodwin & Renne, 1991; Dunlap, 1997) and mylonitic dating (West & Lux, 1993; Freeman et al., 1998; Kligfield, et al., 1986) important results were obtained.

The main goal of the present work is to verify the feasibility of the $^{40}Ar/^{39}Ar$ methodology in the study of tectonic reactivations of shear zones using the new installed Ar-Ar equipment in the Centro de Pesquisas Geocronológicas in the Institute of Geosciences at the University of São Paulo, Brazil. To reach the proposed objectives, deformed, neo-formed and recrystallized minerals were used from igneous and metamorphic rocks of the Romeral fault system in the northern part of the Central Cordillera of Colombia. Fine muscovites and biotites found in mylonitic rocks and in hydrothermal alteration areas generated during the main tectono-magmatic episodes supplied the main keys for obtaining deformational ages. K-Ar analyses in mylonitic, volcanic and hydrothermalized rocks were carried out to yield complementary results.

The main advantage of the method consists on the possibility of obtaining reliable results with little amount of material, such as individual crystals or small mineral concentrates of fine micas carefully selected by handpicking.

## DATING MYLONITIC ROCKS

The knowledge of the deformational textures and the role of the different mineral species in the deformational fabric becomes necessary in order to determine which minerals can supply results with geological meaning. In addition, the understanding of the deformational mechanisms and temperatures reached during the process is necessary to establish that such results represent deformational and not cooling ages. Because of their good Ar retentivity, analyses of pure concentrate of neo-formed potassium micas have a potentiality to yield deformational ages in greenschist facies conditions.

Neo-crystallization of micas during deformation is a widely recognized phenomenon and is the most important process in the Ar-Ar dating, because usually these crystals should not contain $^{40}Ar$ in relevant amount at the moment of deformation (Reddy & Potts, 1999). Micas offer a considerable potential to date crustal deformations because of their stability in a wide spectrum of metamorphic conditions. Therefore, from a practical point of view, the most important limitation to meaningful results is the appropriate concentration of the mineral of interest, so much for the amount of sample available as for its grain size, because the amount of gas to be obtained in the ar extractions should be well above the level of blank of the extraction system.

In this work, strongly deformed and mylonitized rocks with generation of neo-formed potassium micas are key samples to obtain deformational ages. Intensely altered rocks, products of fluid activity, are a second objective for complementary results.

## SAMPLING AND EXPERIMENTAL METHODS

The sampling area for this study is located WSW of Medellin, Colombia within the Romeral fault system. Four metamorphic rocks, six mylonitic rocks and two igneous rock samples were crushed, ultrasonicated in ethanol, and dried. Ten to fifteen single crystals of biotite or muscovite from each sample were picked with metal tweezers, placed in wells in Al-irradiation disks, and irradiated, together with Fish Canyon sanidine fluence monitors, in the IEA-R1 nuclear reactor at IPEN, São Paulo, Brazil. After 2-3 weeks, three grains from each sample or concentrates of 10-15 grains for fine micas were analyzed by the laser incremental heating $^{40}Ar/^{39}Ar$ method at the CPGeo-USP laboratory. Irradiation, analysis, and interpretation procedures follow the methodology presented in Vasconcelos et al. (2001). Additionally, K-Ar whole rock analysis for six mylonitic rocks and three hydrotermalized volcanic rocks for comparison purposes were carried out (Table 1).

The final selection of samples for Ar-Ar analysis was defined during a detailed petrographic study supported by X ray and SEM studies, especially for the finest grained rocks.

## THE PROBLEM OF GRAIN SIZE AND ANALYTICAL ASSESSMENT

Sample size requested for analysis $^{40}Ar/^{39}Ar$ is one of the main advantages of the method, because in deformational areas neo-formed micas are usually very fine, about 25 to 50 μm. The amount and size of the mineral of interest in mylonitic rocks frequently prevent the concentration of enough material for analysis.

The amount of sample necessary for Ar-Ar analysis depends fundamentally of the amount of radiogenic $^{40}Ar^*$

that allows the measurement of the $^{40}Ar/^{39}Ar$ age spectrum with appropriate resolution. The most important factors that rule the generation of enough gas are the age and the concentration of K in the sample.

For the analyses made in this work, the argon extraction line in the $^{40}Ar/^{39}Ar$ equipment at CPGeo-USP reached blank values in the order of $10^{-16}$ moles of $^{40}Ar$, varying between $1.73x10^{-16}$ and $1.40x10^{-15}$ moles, with an average value of $2.79x10^{-16}$ moles of $^{40}Ar$. Individual grains with size among 0.5-1.0 mm and weight between of $10^{-3}$ and $10^{-5}$ gm, yielded values between $1.99x10^{-12}$ and $5.46x10^{-14}$ moles of $^{40}Ar$ for biotite, $1.31x10^{-12}$ and $2.97x10^{-13}$ for muscovite, and $1.21x10^{-13}$ and $2.01x10^{-15}$ for amphiboles. Biotite and muscovite crystals yielded levels of released $^{40}Ar$ up to two magnitude orders higher than blanks.

Concentrates of about 10-15 grains of fine micas from deformed and mylonitic rocks with approximate total weights of $1x10^{-5}$ gm, corresponding to individual grain sizes among 25 to 50 µm, yielded values between $1x10^{-14}$ and $1x10^{-16}$ moles of $^{40}Ar$. Such amount of gas is at the limit of the analytical possibilities of the equipment, and the obtained spectra varied greatly in quality. Some samples could not release gas above blank values. However, most of them yielded enough gas in the initial steps, and the age spectra could be considered geologically meaningful.

The curves of figure 1 represent acceptable minimum values in terms of amount of sample vs. age according to the obtained experimental results. About 50 times the amount of gas in moles of $^{40}Ar$ above the blank of the system was considered acceptable for appropriate determinations. Analyses with slightly lower amounts of gas released didn't yield good quality step heating spectra. Additionally to the minimum values suggested in this work for appropriate analyses, other difficulties related to the size of the material to be analyzed and to the amount of atmospheric argon present in the sample were found. An additional problem is the difficult manipulation of very small grains and the risk of contamination during the operation at the nuclear reactor and in the handling of samples in the holder.

From the results, were confident that a concentrate of 15 to 20 grains of fine micas (25-50 µm.), with an average percentage of K of 7% and older than 10 Ma could yield a reliable age (Fig. 1).

## DEFORMATIONAL AND RESETTING AGES

Mylonitic rocks are direct representatives of activation episodes of fault systems in ductile intermediate crustal levels. Very good spectrums were obtained from mylonitic rocks in neo-formed fine mica concentrates, yielding in most cases concordant plateau ages for different samples and similar K-Ar ages (Table 1). Mineral concentrates were analyzed instead of individual grains because of the fine grain size and the necessity of obtaining enough gas for reliable measurements.

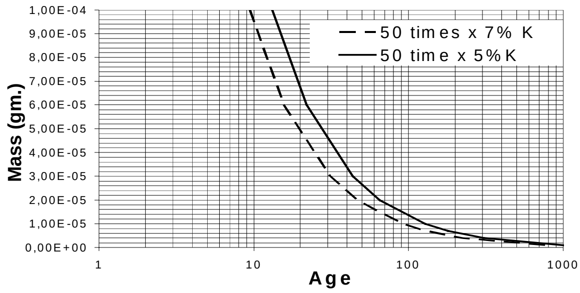


**Figure 1.** Curves representing the acceptable minimun values for appropiate Ar-Ar analysis in terms of amount of sample vs. Age according to the obtained experimental results. Values of 40Ar released 50 times the blank of the system for 7 and 5% K are represented.

Samples collected from the basic volcanic Quebradagrande Fm., correspond to centimetric mylonitic bands within the sequence, presenting variable degree of deformation.

A tuffaceous mylonitic rock of intermediate composition yields a well defined plateau age in sericite above 90% of the total gas released of 74 ± 2 Ma, interpreted as a deformational age because the temperatures reached are probably below 450° C. This age is close to a whole rock K-Ar apparent age of 69 ± 2 Ma for the same sample. Similar results were obtained from a phyllonite with fine grained sericite generation. In spite of the little quantity of gas released, it was possible to obtain an amount of gas critically above the blank value in the first two steps of the degasification process. One of the concentrates yields a plateau age of 72 ± 4 Ma for 80% of gas released, considered as a reliable value, because is concordant to a whole rock K-Ar analysis of 65 ± 3 Ma (Table 1) for the same sample. This age value is interpreted as directly related to the activity of the fault system, indicating a time of deformation. The reached temperatures would be in the interval corresponding to the greenschist facies, given the presence of neoformed chlorite and sericite and formation of ribbons textures in quartz with appreciable recovery, confirming a temperature for deformation compatible with metamorphism of medium to high greenschist grade (Simpson, 1985). The integrated ages around 34 Ma are considered as due of the heterogeneity of the spectrum, without geological significance.

A basic mylonitic rock within the same Quebradagrande formation was analyzed using a concentrate of graphite-bearing sericite grains, given the reduced size of the sericites. The amount of gas obtained was very low, with generation of gas just above blanks value for the initial steps. The obtained spectrum is of low analytical quality and the integrated age of 84 ± 4 Ma and the K-Ar whole rock age of 66 ± 3 Ma (Table 1) barely reflect the deformational episode of the upper Cretaceous, recorded for some others regional samples. A sample of more mafic composition in the same volcanic rocks shows a high homogeneity for 80% of gas released and yields biotite plateau ages of 89.8 ± 1 Ma and 87.8 ± 0.7 Ma, considered as cooling ages of an Albian-Cenomanian

**Table 1.** Ar-Ar and K-Ar results of deformed and hydrothermalized rocks.

| Unit | Mineral | Integrated Age (Ma) | Plateau Age (Ma) |
|---|---|---|---|
| Tuffaceous mylonitic rocks of Quebradagrande formation | Ser | 86±2 | 74±2 |
| | | 84.9±1.3 | 81±5 |
| | Bi | 96.6±1.1 | 89.8±1.1 |
| | | 83.2±0.7 | 87.8±0.7 |
| | Ser | 93±14 | |
| | | 89±5 | |
| Sabaletas schist | Ser | 102±3 | |
| | | 115.9±1.6 | 127.5±2.0 |
| Phylonite of Quebradagrande formation | Ser | 32±5 | 72±4 |
| | | 34±15 | |
| Mylonite basic rock of Quebradagrande formation | Ser | 84±4 | |
| | | 70±30 | |
| Santa Barbara metagabbro | Amp | 119±5 | |
| | | 141±7 | |
| Sabanalarga batholith | Amp | 152.7±0.7 | |
| | | 145.9±0.5 | |
| | Amp | 145.1±0.7 | |
| | | 90±20 | 89.9±0.6 |
| | Amp | 92±3 | 92±2 |
| | | 108±4 | 106±3 / 95±4 |
| Palmitas gneiss | Bi | 67.95±0.11 | 68.13±0.12 |
| | | 68.09±0.14 | |
| Amphibolite of the Arquia group | Bi | 4.5±0.4 | 5.6±0.4 |
| | | 0.3±1.1 | 4.5±1.1 |

| Unit | Material | %K | $\%^{40}Ar$ Atm | Age (Ma) |
|---|---|---|---|---|
| Gabbro Quebradagrande formation | WR | 0.20 | 56.45 | 102±7 |
| | WR | 0.94 | 34.19 | 64.7±3 |
| Tuffaceous mylonitic rocks of Quebradagrande formation | WR | 1.73 | 19.66 | 73±2 |
| | WR | 4.04 | 12.49 | 62±1 |
| | WR | 1.95 | 18.19 | 69±2 |
| Basic pyroclastic rock of Quebradagrande formation | WR | 0.31 | 58.58 | 68±5 |
| Mylonite básic rock of Quebradagrande formation | WR | 1.08 | 39.82 | 66±3 |
| Phylonite of Quebradagrande formation | WR | 0.59 | 47.67 | 65±3 |
| Mylonitic band bordering metric unaltered lenses of Triassic quartzodioritic material of the Cambumbia stock | WR | 0.59 | 42.66 | 86±16 |

rock deformation episode. However, a whole K-Ar age of 62 ± 1 Ma was obtained for the same sample, clearly discordant in relation to the previous results, suggesting that this age is not well constrained. To complete the picture, a tuffaceous mylonitic rock of felsic composition collected in the same outcrop yields a very heterogeneous spectrum. The amount of gas obtained for this sample was critical, and it was possible to obtain an amount of gas above blank levels only in the first stages of fusion.

The ages obtained when about the 90% of the gas was released, are considered as hardly reflecting a not well constrained regional deformational episode about 100 Ma. Integrated ages of and 89 ± 5 Ma are considered as a reasonable approach for this deformational episode, and they agree within experimental errors with values registered for other samples in the same area. However, were obtained K-Ar whole rock discordant results of 73 ± 2 Ma suggesting again that the age is not very well constrained.

One sample from the Sabaletas quartz micaceous polymetamorphic schist yielded a sericite plateau age of 127.5 ± 2.0 Ma registered when the largest amount of gas was released. This age coincides in general with values obtained for the concentrate of a second analyzed mineral of the same sample. A precise age was not obtained, and the plateau age is here considered to be close of the deformational metamorphic event that formed the sericites.

Analyses for the Palmitas gneiss show very well defined plateaus, indicative of a complete resetting of the system because the complete recrystallization of biotite. The plateau age obtained of 68.13 ± 0.12 Ma may reflects the intrusion of the Antioquian batholith and or the upper cretaceous well constrained deformational event.

A deformed amphibolite of the Arquia Group, relatively altered, yields very low amount of gas and spectra with high associated error and complex geometries. However, one of the analyzed grains yielded a plateau age in biotite for 50% of released gas of 5.6±0.4 Ma, interpreted as a reflex of the deformation that would have recrystallized the biotites, being coincident with the intrusion ages of neo-Tertiary bodies in the proximities.

## CONCLUSIONS

The most important advantages of the use of the $^{40}Ar/^{39}Ar$ methodology in dating deformational episodes are: (1) the possibility to work with potassium micas, which commonly define deformational fabrics; (2) the use of a small amount of sample and (3) the possibility to interpret adequately the spectrum obtained.

In order to obtain satisfactory results we shall observe: (1) the usefulness of working with highly deformed and highly hydrothermalized rocks with neoformed potassium micas and (2) the need to carefully isolate micas from the most deformed segments of the hand sample. Finally, complementary K-Ar whole rock analyses in the case of fine grained, hydrothermalized and mylonitic rocks may yield important information.

An amount of 50 times of $^{40}Ar$ moles above the system blank was considered acceptable as a lower limit for reliable analysis, provided that the amount of atmospheric argon in the extraction system be sufficiently low.

Concentrates with 15 to 20 grains of fine micas with K concentration of 7% and grainsize between 25 to 50 µm, may yield significant results for samples older than 10 Ma.

For fine micas from mylonitic rocks, with grain size between 25 to 50 µm, between $1x10^{-14}$ e $1x10^{-16}$ moles of $^{40}Ar$ were obtained at the limit of the analytical possibilities of the equipment. Most samples yielded enough gas for analysis in the initial steps of the fusion process, where it was possible obtain geologically meaningful results.

The strong concordance observed between K-Ar whole rock ages and mineral Ar-Ar results from the same samples, and samples of the same area, indicate that the results obtained have geological meaning and show that Ar-Ar analysis for small concentrates of potassium micas yield good results.

Lower Cretaceous deformational events were recorded in a quarzo-sericite schist, hydrothermal ages of the Sabanalarga batholith and amphiboles from a metagrabbro.

On the basic volcanic sequences of Quebradagrande formation of lower Cretaceous age, a K-Ar total rock age of 102 ± 7 Ma in a cataclastic gabbro was interpreted as hydrothermal alteration age indicative of the fault system activity in Albian – Cenomanian times. $^{40}Ar/^{39}Ar$ analyses from the same sequence yielded biotite and sericite plateaus ages between 87 and 90 Ma of poor interpretative value.

In the same volcanic sequence were obtained apparent Ar-Ar plateau ages about 72 Ma very well constrained with whole rock K-Ar results, being this the better recorded deformational event. Another whole rock K-Ar ages between 66 and 73 Ma confirm the lower cretaceous deformational event. $^{40}Ar/^{39}Ar$ biotite plateau ages of 5.6±0.4 Ma obtained for an amphibolite, recorded the last important activation episode of the Fault System.

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