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Automated mapping of K-feldspar by electron backscatter diffraction and application to $^{40}Ar/^{39}Ar$ dating

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ABSTRACT

The ability to quantify feldspar microstructure using the electron backscatter diffraction (EBSD) method has direct application in the study of rock deformation and strain kinematics. However, automated EBSD analysis of low symmetry phases, such as feldspar, has previously proven difficult. Here, we successfully apply the EBSD method to a number of granitic feldspars and develop automated phase and orientation mapping to discriminate K-feldspar and plagioclase, and quantify orientation variations within individual K-feldspar grains. These results represent the first automated quantitative mapping of orientation microstructure in K-feldspar. We use the method to evaluate the relationship between microstructure and $^{40}Ar/^{39}Ar$ age, a controversial problem in thermochronology. In a granitic K-feldspar from central Australia, the range of observed orientation domains matches the small to intermediate and largest domain sizes predicted from multiple-diffusion domain modeling. In situ ultra-violet laser microprobe analyses show that the youngest ages from the $^{40}Ar/^{39}Ar$ age spectra are recorded by grain mosaic K-feldspars with diameter around 10–50 μm. These K-feldspars are the smallest coherent microstructural features observed on scales of >1 μm. Large 250–1000 μm diameter microstructurally simple grains record the oldest ages observed in the age spectrum. These results suggest a first order relationship between K-feldspar microstructure and $^{40}Ar/^{39}Ar$ age and demonstrate a microstructural control on multidomain diffusion.

1. Introduction

Electron backscatter diffraction (EBSD) is a scanning electron microscope (SEM) technique that allows phase identification and the quantitative analysis of orientation variations in crystalline materials. The method utilizes diffraction of electrons by the crystalline lattice, which generates a number of bands ("Kikuchi" bands) that each corresponds to a set of lattice planes with a width that is directly related to lattice spacing (Randle, 2000). Together these bands form an electron backscatter diffraction pattern (EBSP) that is characteristic of both the phase and orientation of the crystal (e.g., Prior et al., 1999). By automatically collecting EBSPs over a predefined grid, EBSD data can be used to generate maps of phase and orientation data that allow the linkage of EBSD data to spatial position on a particular surface within the sample. Such an approach is performed on specially polished material surfaces and is non-destructive, allowing additional analytical techniques to be applied to the same sample. This approach therefore has certain advantages over much higher spatial resolution transmission electron microscopy (TEM) for investigating the relationship between microstructure and geochemistry. Unlike transmission electron microscopy, EBSD analysis can also be coupled directly with orientation contrast imaging (Prior et al., 1996), providing constraints on the microstructural context.

EBSD analysis of high symmetry geological materials such as olivine (Faul and Fitz Gerald, 1999), garnet (Prior et al., 2002), calcite (Bestmann and Prior, 2003), galena (Skrotzki et al., 2000) and zircon (Reddy et al., 2007) has yielded useful insights into the microstructural behavior of these minerals during recrystallization, deformation and/or grain growth. However, EBSD analysis of lower symmetry phases, and particularly feldspars, has proven difficult. The reasons for this difficulty are the complex nature of feldspar EBSPs, the similarity of EBSPs between different feldspar phases, the various feldspar twin laws, pseudosymmetry in feldspars and problems associated with sample preparation. As a result automated EBSD analysis of feldspar has been difficult and the successful application of EBSD to feldspar (e.g. Prior and Wheeler, 1999; Jiang et al., 2000) has relied upon manual indexing of EBSPs, which is time consuming and does not readily permit the integration of geochemical data within a spatially-constrained microstructural context afforded by automated EBSD mapping.

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Here, we develop the use of automated EBSD mapping of alkali-feldspar in a variety of granitic rocks. We present a description of the method, operating conditions and indexing parameters employed. We then show the results of the automated EBSD mapping, including: (1) the successful discrimination of alkali-feldspar and coexisting plagioclase; and (2) the collection and analysis of quantitative crystallographic orientation data for K-feldspars.

We apply the EBSD method to the problem of linking quantitative orientation microstructure and $^{40}Ar/^{39}Ar$ age in K-feldspars. The mineralogy of K-feldspar has been extensively studied and its use in thermometry (e.g. Elkins and Grove, 1990) and argon geochronology (e.g. Spell et al., 1996; Swisher et al., 1993; McDougall, 1985) is well established. However, the thermochronologic use of K-feldspar remains controversial. The multiple-diffusion domain (MDD) model (Lovera et al., 1989; Richter at al., 1991) attests that argon loss in K-feldspars is controlled only by thermally-activated volume diffusion and that the strong $^{40}Ar/^{39}Ar$ age gradients often seen in K-feldspars are the result of variable argon retention by diffusion domains of different sizes (Lovera et al., 1989, 1991). The notion of a number of different sized diffusion domains comes largely from the characteristic non-linear Arrhenius behavior (Lovera et al., 1989). Individual diffusion domains are non-interacting and of simple geometry, and argon is lost instantaneously from domain boundaries. The fundamental tenet of the model is that the retention of argon during cooling in nature and the loss of argon during step-heating in the laboratory is controlled only by thermally-activated volume diffusion. The presence of a range of diffusion domain sizes yields a range in closure temperature that can be inverted to yield continuous cooling histories (Richter et al., 1991). As such, the method has become a potentially powerful tool in the reconstruction of exhumation histories and in the solution of various tectonic and structural geology problems (e.g. Dunlap and Fossen, 1998; McLaren et al., 2002) where it appears to give geologically reasonable cooling histories that are internally consistent and that are also consistent with apparent ages from higher and lower temperature chronometers, such as $^{40}Ar/^{39}Ar$ muscovite ages and apatite fission track ages.

However, only about half of all the K-feldspars analysed are suitable for thermal history analysis (Lovera et al., 2002) and there is considerable controversy regarding the validity of the method. In particular: (1) Lee (1995) questioned the assumption that volume diffusion is the only mechanism of argon loss, arguing that fast-pathway diffusion can also influence the argon release; comparisons of UV laserprobe Argon data with qualitative analysis of deformation microstructure support this (Reddy et al., 2001); (2) Parsons et al. (1999) questioned the presence of a discrete domain structure with the specific characteristics required by the MDD model as well as the assertion that K-feldspar microstructures form only at temperatures above the closure temperature of diffusive argon loss; and (3) the role of sub-micron features such as micropores, subgrain boundaries and 'nanotunnels' remains unresolved (Fitz Gerald et al., 2006).

Central to all of these arguments is the question of how microstructure and argon loss are linked and to some extent this controversy reflects the inconsistency between the complex microstructural characteristics observed in K-feldspar and the relative simplicity of the MDD model. The work of Reddy et al. (2001) indicates that the way in which strain is accommodated within K-feldspar is a key control on the way in which the distribution of argon is modified. However, despite more than 20 years of work characterizing textural variations in K-feldspar, particularly at the sub-micron scale, there is still no explanation for the correlation between argon age and deformation-related microstructure reported by Reddy et al. (1999, 2001) other than the observation that orientation domain boundaries facilitate the grain-scale redistribution of argon. To help resolve this problem it is essential to integrate quantitative analysis of intragrain orientation variations with thermochronologic data. We link orientation microstructure and $^{40}Ar/^{39}Ar$ age by analysing the K-feldspar from a given sample using both $^{40}Ar/^{39}Ar$ step-heating (on separated K-feldspar grains) and in situ $^{40}Ar/^{39}Ar$ analysis (on individual K-feldspars in thin section). As such, this study builds on previous work investigating deformation-related subgrains and Ar isotope systematics (Reddy et al., 1999, 2001) by providing the first link between *quantitative* orientation data, derived by EBSD, and $^{40}Ar/^{39}Ar$ ages.

2. Sample descriptions

Alkali-feldspar from three granitic sample suites was selected for this study. In all cases, macroscopically undeformed feldspars from undeformed granites were analysed to better enable results to be directly linked to samples typically used in the MDD approach. Compositionally, the analysed feldspars range from grains, which are obviously perthitic under the light microscope, to homogenous microcline or orthoclase grains.

2.1. Big Lake Suite granites, Warburton Basin, Australia

The Big Lake Suite granites are of Carboniferous age (323 ± 5 and 298 ± 4 Ma; Gatehouse et al., 1995), and intrude the Warburton Basin at the base of the Cooper/Eromanga Basin in northern South Australia. The granites are compositionally and texturally complex and three samples exhibiting a variety of textural characteristics were chosen. These samples were obtained from core material extracted from three petroleum exploration wells; Sample 02-149 from a depth of 2895.2 m in Moomba-1; Sample 01-147 from a depth of 3056.7 m in Big Lake-1 and 02-152 from a depth of 3748.8 m in McLeod-1. Uncorrected temperatures in the granite range from 160 to 230 °C, and at least in part represent a recent increase in geothermal gradient associated with high temperature fluid-flow (McLaren and Dunlap, 2006). Sample 02-149, containing K-feldspar, plagioclase, quartz and biotite, is the most pristine of the three samples. In hand specimen it is characterized by classic igneous textures and highly lustrous euhedral crystal faces. Individual alkali-feldspar grains are coarsely perthitic, show good crystal shape, and an almost total absence of alteration features such as clay minerals or dissolution pits (McLaren and Dunlap, 2006). Sample 02-147 shows complex textural features on the scale of individual grains and also complex grain boundary zones; the feldspars are characterized by moderate development of 10–50 μm clay mineral laths and perthitic exsolution textures. In hand specimen the sample is characterized by sugary-textured opaque feldspar. The third sample (02-152) exhibited extremely complex textural features with feldspar grains characterized by large overgrowths of highly altered mica and clay minerals, probably as a result of extensive hydrothermal alteration and/or recrystallization. This sample was unable to be polished to sufficiently high quality for EBSD analysis to be performed.

The alkali-feldspars in all three samples are almost pure orthoclase containing only very minor amounts of Na (<1.6 wt%) and Ca (<0.04 wt%). The average of a number of point analyses give alkali-feldspar compositions in the range $An_{0-0.1}Ab_{4.3-8.2}Or_{91.7-95.7}$ (Table 1). Coexisting plagioclase in perthitic exsolution lamellae is almost pure albite with a composition around $An_{3.2}Ab_{95.5}Or_{1.3}$ (Table 1). The granites are inferred to have intruded during compression associated with the Alice Springs Orogeny (Sun, 1997) and the thermal conditions associated with this event in the region suggest that the granites are likely to be intermediate temperature melts, with crystallization temperatures ~700–750 °C (Sun, 1997; McLaren and Dunlap, 2006).