Thermal Overprinting of Natural Remanent Magnetization and K/Ar Ages in Metamorphic Rocks

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Burial metamorphism generates secondary components of NRM (natural remanent magnetization). Often two or more remanences are found superimposed in a single geological unit. We trace the steps necessary to resolve and date such magnetic overprints in three studies of intensely metamorphosed rocks from the Grenville Province of the Canadian Precambrian Shield. First, it is shown that the various components may be associated with different rock-forming minerals and can be separated by orienting aggregates of dark or light grains in coarse-grained samples. Next, these magnetizations are shown to have originated at least in part as TRM's (thermoremanent magnetizations). Their relative ages can then be assigned on the basis of blocking temperatures. Finally, we estimate their absolute ages using the $^{40}\text{Ar}/^{39}\text{Ar}$ stepwise heating technique, taking into account the effects of a long heating-cooling history on both the magnetic and radiometric systems.

1. Introduction

Recent paleomagnetic studies of North American Precambrian rocks (e.g., BUCHAN and DUNLOP, 1973, 1976; IRVING *et al.*, 1974; FAHRIG *et al.*, 1974; UENO *et al.*, 1975; ROY and LAPOINTE, 1976; IRVING and MCGLYNN, 1976; SCHWARZ, 1977; VITORELLO and VAN DER VOO, 1977; BUCHAN, 1977a) have revealed numerous examples of superimposed natural remanent magnetizations (NRM's). One of the component NRM's may be primary, but the other component(s) are thermal or chemical overprints resulting from burial metamorphism. Frequently, all the components are overprints.

Paleomagnetic techniques for separating multiply overprinted NRM's are just being developed. Determining reliable paleomagnetic directions for the components—a difficult task if their coercivity and blocking temperature spectra overlap—is not enough. Determining the origins of the components, whether TRM or CRM, is an obligatory first step in assigning relative, and ultimately absolute, ages to the components. Age determination is almost taken for granted in single-component studies where a primary NRM can be assumed, but it is far from trivial in rocks that have been repeatedly remagnetized. Indeed it is the principal difficulty in establishing apparent polar wander paths (APWP's) for Precambrian time.

This paper describes some novel approaches to the problems of metamorphic

Fig. 1

overprinting. Our examples are drawn from the Grenville structural province of the Canadian Precambrian Shield, where regional metamorphism was unusually severe, and uplift and cooling were unusually slow, spanning 100 to 200 my. Grenville rocks frequently exhibit superimposed NRM's in individual samples.

As our case histories will make clear, a complete multi-component paleomagnetic study demands time and ingenuity. On the other hand, a successful study of this kind determines an APWP in miniature for the body studied, rather than a single paleopole. In what follows, magnetic studies are due to K.L.B., M.O.McW and D.J.D. and $^{40}\text{Ar}/^{39}\text{Ar}$ work is by G.W.B. and D.Y.

2. Resolving Superimposed Magnetizations by Bulk Mineral Separation

The isolation of magnetic components superimposed in individual specimens has usually been attempted using the indirect approaches of vector subtraction (ZIJDERVELD, 1967; BUCHAN and DUNLOP, 1976) and the intersection of remagnetization circles (HALLS, 1976). We are currently attempting a direct separation of components in samples of the coarse-grained Bark Lake diorite (one of the Haliburton intrusions of BUCHAN and DUNLOP, 1976), employing magnetic microanalysis (WU *et al.*, 1974).

Consider, for example, the sample of Fig. 1. In whole-rock analysis (Fig. 1a), the direction of magnetization moves progressively on both alternating field (AF) and thermal demagnetization from a shallowly inclined Grenville *B* direction toward a steep *A* direction. (The *A* and *B* directions for the Haliburton intrusions were established by BUCHAN and DUNLOP (1976) utilizing stable endpoints and vector subtraction respectively.) In Fig. 1a, directional coherence diminishes at the higher alternating fields, presumably because the *A* component constitutes a small fraction (7%) of the NRM intensity (1.8×10^{-3} emu/cm³).

The sample of Fig. 1 is composed of rather large aggregates of dark rock-forming minerals (largely hornblende and biotite, the latter often altered to chlorite) set in a groundmass of both light (feldspar) and dark (biotite, hornblende and pyroxene) minerals. We oriented blocks of these bulk mineral fractions whose magnetizations are sufficiently intense to be measured with a standard spinner magnetometer. These separates are 0.1 to 2 cm³ in volume, larger than the oriented grains cut from thin sections by WU *et al.* (1974).

We find that the *A* and *B* magnetizations are preferentially associated with the different mineral fractions of the whole rock. The *B* component (Fig. 1c) is concentrated in the large aggregates of dark minerals where titanomagnetites are observed

Fig. 1. AF and thermal demagnetization of a sample of the Bark Lake diorite (one of the Haliburton intrusions) showing superimposed *A* and *B* magnetizations. Directions are plotted on a stereographic projection. Closed and open symbols indicate positive and negative inclinations respectively. Circles and crosses denote AF results, triangles denote thermal results. Numbers indicate peak AF's in Oe or temperatures in °C. Characteristics of (a) a whole-rock specimen, and oriented bulk separates from (b) groundmass material and (c) a dark mineral segregate are compared.

to be the dominant opaque mineral. The *A* component, on the other hand, resides preferentially in the groundmass fraction where both magnetite and hematite have been identified in polished thin section.

The *A* component intensity (Fig. 1b) constitutes nearly 70% of the NRM (0.66×10^{-3} emu/cm³) of the groundmass separate. The *A* endpoint direction is now attained 100 Oe, whereas the magnetization vector of the whole-rock sample had barely begun to swing in 100 Oe.

No evidence of an *A* component is observed in the dark mineral segregate (Fig. 1c). The magnetization direction remains constant to 150 Oe, then moves to somewhat higher declinations, before becoming random at 600 Oe and above. The directional shift between 150 and 400 Oe may be due, in part, to problems encountered in measuring the low-intensity magnetization of the small separate.

Nevertheless, this example demonstrates the usefulness of direct mineral separation to isolate the directions of superimposed magnetizations in coarse-grained rocks. This method is applicable to several problems in the study of multi-component magnetizations:

- a) confirming whole-rock endpoints which may have been established by indirect methods such as vector subtraction and intersection of remagnetization circles;
- b) isolating endpoints when indirect methods fail; and
- c) determining the magnetic mineral carriers and their association with rock-forming minerals.

This latter application is potentially important in establishing the origin and relative ages of superimposed components.

3. Problems in Establishing Relative Ages of Superimposed Magnetizations

Isolating endpoint directions constitutes but the first step in the study of multi-component remanences. Next, one must date each component. We will consider this problem in two steps: first, establishing the *relative* ages of the various components by determining their origins, and second, establishing their *absolute* ages by radiometric dating.

3.1 *Relative ages of thermal and chemical overprints*

There is a widely-held misconception that relative ages of superimposed magnetizations can be defined more or less automatically by examining their blocking temperature (T_B) spectra. The component with the highest blocking temperatures is deemed to be the oldest, while successively lower T_B components are assumed to be progressively younger.

This simple picture presupposes that all components are partial TRM's, primary or secondary. A minimum test of this assumption is that, in comparing samples from the same site or neighbouring sites, the blocking temperature ranges of the various components should not overlap. Even with this proviso, it is easy to imagine anomalies, e.g., a CRM overprint carried by hematite with apparent blocking temperatures

(apparent, because the overprint is chemical, not thermal) between, say, 600 and 670°C that postdates a magnetite TRM with $T_B \leq 580^\circ\text{C}$. Blocking temperature data alone are not sufficient to prove the thermal origin of overprints.

3.2 Nature and relative ages of Grenville components

The necessity for detailed magnetic studies to establish relative ages is illustrated by the Haliburton (BUCHAN and DUNLOP, 1976) and Thanet (BUCHAN, 1977a) intrusives. In each study, a steeply inclined Grenville *A* direction and a shallow *B* direction have been isolated (Fig. 2). Note that the *B* directions are of the same polarity in the two intrusives but the *A* directions are reversals of each other, being steeply upwards in the Haliburton and steeply downwards in the Thanet.

The Haliburton magnetization vector swings from the *B* towards the *A* direction on both AF and thermal demagnetization (Fig. 2a). Other published Grenville studies (IRVING *et al.*, 1974; UENO *et al.*, 1975) show the same pattern, which has been taken to be characteristic. On a simple thermal overprinting model, the *A* magnetization, since it has higher T_B 's, is older than the low T_B *B* component.

However, results from the Thanet gabbro do not agree (Fig. 2b). Upon AF demagnetization, the directional swing is from *B* towards *A*, but on thermal demagnetization, the vectors always move away from the *A* direction towards the *B* direction. That is, the *B* component, although it is 'soft' relative to the *A* component upon AF demagnetization, has higher blocking temperatures than the *A* component.

If we assume that the Haliburton *A* and Thanet *A* components are essentially contemporaneous, as are the Haliburton *B* and Thanet *B* magnetizations, the assumption of thermal overprinting cannot be correct in both formations. Additional evidence is necessary to assign relative ages to Grenville *A* and *B* components.

Fig. 2. Comparison of AF and thermal demagnetization characteristics of samples showing superimposed (*A+B*) magnetizations from (a) the Haliburton intrusions and (b) the Thanet gabbro. Symbols as in Fig. 1. Best-fitting great circle paths are superimposed on the data points.

3.2.1 The A magnetization

At different sampling sites of the Haliburton intrusions, the high- T_B A component has T_B 's near 580°C consistent with magnetite as the dominant magnetic carrier, and T_B 's well above 600°C, implying that hematite carries the remanence.

Unmistakable evidence that the high- T_B A component is carried by both magnetite and hematite, even within individual samples, is found in the Magnetawan metasediments (McWILLIAMS and DUNLOP, 1975). Figure 3 shows a set of recently measured thermal demagnetization curves for Magnetawan samples that contain purely the A component. The closely spaced heating steps leave no doubt that although the bulk of the A blocking temperatures cluster near 580°C, the Curie point of magnetite, a significant fraction of the A magnetization has blocking temperatures

Fig. 3. Thermal demagnetization characteristics of typical A -remanence samples from the Magnetawan metasediments. The curves have been stacked to spread them. The Curie temperatures of magnetite (580°C) and hematite (670°C) are shown as dashed lines. All inclinations are upwards and all points represent thermal data. Sample numbers are in italics.

up to 650–670°C. In other words, magnetite and hematite, coexisting within the same hand-sample, both carry an *A* magnetization.

It is difficult to envisage a low-temperature chemical process that would generate *both* magnetite and hematite at the same time (the time recorded by the *A* geomagnetic field direction). It is possible that the hematite fraction of the *A* magnetization is a CRM, but the magnetite fraction, at least, must be a TRM. The *A* component has *true* blocking temperatures of 550–580°C and possibly higher ones as well.

3.2.2 The *B* magnetization

The Haliburton *B* magnetization contains two fractions (BUCHAN and DUNLOP, 1976). One fraction has T_B 's > 450°C. It is undoubtedly of chemical origin since its highest blocking temperatures frequently overlap those of the *A* component.

The other fraction has $100^\circ\text{C} < T_B < 450^\circ\text{C}$. Because of its similarity to artificial low-temperature partial TRM's produced in the same samples (BUCHAN, 1977b), we believe it is a low-temperature thermal overprint. IRVING *et al.* (1974) independently reached the same conclusion about the origin of the 'secondary' component of NRM in the Morin anorthosite.

The very low blocking temperatures of the Haliburton *B* component rule out any reheating subsequent to the acquisition of the *B*. The high- T_B *A* component necessarily predates the low- T_B *B* component. If the Thanet *A* and *B* components date the same overprinting events as the Haliburton *A* and *B* components respectively, the Thanet *B* magnetization is a chemical overprint, with high *apparent* blocking temperatures.

4. Techniques for Establishing Absolute Ages of Superimposed Magnetizations

Once it has been shown that one or more of the components in a multi-component study is of thermal origin, one can proceed to the second stage of the dating problem: estimating the absolute age of each thermal component.

First we date the body radiometrically, obtaining whole-rock and mineral-separate ages. For example, the $^{40}\text{Ar}/^{39}\text{Ar}$ stepwise heating technique gives two distinctly different ages for the Bark Lake (Haliburton) intrusion (Fig. 4): approximately 990 my for hornblende, and about 920 my for biotite. The reason for this apparent discrepancy is probably that these minerals became closed to Ar diffusion at different closure or blocking temperatures during the slow cooling that followed burial and metamorphism of the region.

Indeed, with a sufficient number of reliable age determinations from minerals with a wide range of closure temperatures, we can define an approximate temperature-time curve for the body. Magnetic blocking temperatures of the various partial TRM components can then be matched with this calibrated cooling curve to date the magnetizations.

Blocking temperatures in nature during slow cooling differ from blocking temperatures determined in rapid laboratory heatings. Hence, we must always take into

Fig. 4. Hornblende and biotite $^{40}\text{Ar}/^{39}\text{Ar}$ age determinations for the Bark Lake diorite of the Haliburton intrusions. The length of the bars represents the volume fraction of Ar released at a given temperature (in hundreds of degrees Celsius). The height of the bars represents 1σ analytical error including all sources except error in the apparent age of the reference mineral and in the decay constants.

account the effects of a long heating-cooling history on both radiometric closure temperatures and magnetic blocking temperatures.

The time dependence of T_B is well known in the magnetic case. For example, PULLAIAH *et al.* (1975) show that, according to the NÉEL (1949) theory of independent single-domain grains, a grain which is just unblocked at temperature T_{B1} in a laboratory-scale time t_1 , will be unblocked in nature over a long time t_2 at a lower temperature T_{B2} :

$$\frac{T_{B2}}{J_S(T_{B2})H_C(T_{B2})} = \frac{\log(Ct_1)}{\log(Ct_2)} \frac{T_{B1}}{J_S(T_{B1})H_C(T_{B1})},$$

J_S being spontaneous magnetization, H_C microscopic coercive force, and C the characteristic frequency of thermal fluctuations ($\sim 10^9 \text{ sec}^{-1}$). Laboratory blocking temperatures are readily extrapolated to geological blocking temperatures using this equation or its graphical representation. Type curves are given by PULLAIAH *et al.* (1975) for Fe_3O_4 and $\alpha\text{Fe}_2\text{O}_3$ and by DUNLOP and HALE (1977) for $\text{Fe}_{2.4}\text{Ti}_{0.6}\text{O}_4$.

Consider the partial TRM components of the Haliburton intrusions. We estimate that the A magnetization carried by magnetite was acquired at temperatures within about 10°C of those observed in the laboratory ($550\text{--}580^\circ\text{C}$) while tempera-

tures of no more than 250°C are necessary to explain B T_B 's of up to 400°C if the B remanence is carried by a low-titanium titanomagnetite.

The diffusion of argon from a rock-forming mineral also depends on a thermal activation mechanism. Accordingly, a grain from which a certain fraction of argon outgasses in a laboratory heating at temperature T_1 for a time t_1 , will lose the same argon fraction in a geological time interval t_2 at a lower temperature T_2 :

$$\frac{1}{T_2} = \frac{1}{T_1} - \frac{R}{E} \log \left(\frac{t_1}{t_2} \right),$$

R being the gas constant and E experimental activation energy.

Alternatively, the time-dependent history of a rock can be defined by considering that the sample was cooling continuously. DODSON (1973) derived an equation for radiometric blocking temperature:

$$(T_B)_{\text{radiometric}} = E/[R \log (A\tau D_0/a^2)],$$

a being a characteristic dimension for the grain, A a geometrical factor, D_0 the diffusion coefficient and τ a time constant related to cooling rate. In practice, the closure temperature is insensitive to uncertainties in τ (see DODSON, 1973), making this method promising for the study of Precambrian shields where cooling rate has been rather slow following burial metamorphism.

In an analogous fashion, YORK (1977a, b) is currently examining magnetic blocking temperatures in a continuously cooling orogen.

We have assumed a slow-cooling model (5°C/my near the blocking temperature) to interpret the Argon data from Bark Lake (Haliburton) rocks shown in Fig. 4, since the presence of well-defined plateaus in the coexisting biotite and hornblende probably restricts any reheating episode to temperatures less than 200–300°C (e.g., BERGER, 1975). We estimate the hornblende and biotite of Fig. 4 to have closed to Argon diffusion at 620 ± 40 (1 σ) and about 225°C respectively. Unfortunately Dodson's formula could not be applied to the biotite data so we quote a geological estimate (TURNER and FORBES, 1976). We emphasize that these results are preliminary and details of the Argon work will be published separately.

The estimated closure temperatures of 620°C for hornblende and 225°C for biotite suggest that the hornblende and biotite ages of 990 and 920 my, likely bracket the acquisition times of the Haliburton A and B magnetizations.

5. Conclusions

Many of the techniques necessary for resolving metamorphically overprinted magnetizations and establishing their relative and absolute ages have yet to be fully developed. Nevertheless, as the case histories of Grenville rocks described in this paper demonstrate, these problems may be solved by a systematic investigation of the distribution of various magnetic components in different rock-forming minerals, the thermal and/or chemical origin of NRM, and the variation of radiometric and magnetic closure or blocking temperatures with time.

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REFERENCES

- BERGER, G.W., $^{40}\text{Ar}/^{39}\text{Ar}$ step heating of thermally overprinted biotite, hornblende and potassium feldspar from Eldora, Colorado, *Earth Planet. Sci. Lett.*, **26**, 387–408, 1975.
- BUCHAN, K.L., Magnetic overprinting in the Thanet gabbro complex, 1977a (in preparation).
- BUCHAN, K.L., Rock magnetic and paleomagnetic studies of multi-component remanences in metamorphosed rocks of the Grenville Province of the Canadian Precambrian Shield, Ph. D. thesis, University of Toronto, Toronto, 1977b.
- BUCHAN, K.L. and D.J. DUNLOP, Magnetization episodes and tectonics of the Grenville Province, *Nature Phys. Sci.*, **246**, 28–31, 1973.
- BUCHAN, K.L. and D.J. DUNLOP, Paleomagnetism of the Haliburton intrusions: Superimposed magnetizations, metamorphism and tectonics in the late Precambrian, *J. Geophys. Res.*, **81**, 2951–2967, 1976.
- DUNLOP, D.J. and C.J. HALE, Simulation of long-term changes in the magnetic signal of the oceanic crust, *Can. J. Earth Sci.*, **14**, 716–744, 1977.
- DODSON, M.H., Closure temperature in cooling geochronological and petrological systems, *Contrib. Mineral. Petrol.*, **40**, 259–274, 1973.
- FAHRIG, W.F., K.W. CHRISTIE, and E.J. SCHWARZ, Paleomagnetism of the Mealy Mountain anorthosite suite and of the Shabogamo gabbro, Labrador, Canada, *Can. J. Earth Sci.*, **11**, 18–29, 1974.
- HALLS, H.C., A least-squares method to find a remanence direction from converging remagnetization circles, *Geophys. J. R. Astron. Soc.*, **45**, 297–304, 1976.
- IRVING, E. and J.C. MCGLYNN, Polyphase magnetization of the Big Spruce Complex, Northwest Territories, *Can. J. Earth Sci.*, **13**, 476–489, 1976.
- IRVING, E., R.F. EMSLIE, and H. UENO, Upper Proterozoic paleomagnetic poles from Laurentia and the history of the Grenville structural province, *J. Geophys. Res.*, **79**, 5491–5502, 1974.
- MCWILLIAMS, M.O. and D.J. DUNLOP, Precambrian paleomagnetism: Magnetizations reset by the Grenville orogeny, *Science*, **190**, 269–272, 1975.
- NÉEL, L., Théorie du trainage magnétique des ferromagnétiques en grains fin avec applications aux terres cuites, *Ann. Géophys.*, **5**, 99–136, 1949.
- PULLAIAH, G., E. IRVING, K.L. BUCHAN, and D.J. DUNLOP, Magnetization changes caused by burial and uplift, *Earth Planet. Sci. Lett.*, **28**, 133–143, 1975.
- ROY, J.L. and P.L. LAPOINTE, The paleomagnetism of Huronian red beds and Nipissing diabase; Post-Huronian igneous events and apparent polar path for the interval –2300 Ma to –1500 Ma for Laurentia, *Can. J. Earth Sci.*, **13**, 749–773, 1976.
- SCHWARZ, E.J., Depth of burial from remanent magnetization: the Sudbury irruptive at the time of diabase intrusion (1250 Ma), *Can. J. Earth Sci.*, **14**, 82–88, 1977.
- TURNER, D.L. and R.B. FORBES, K-Ar studies in two deep basement drill holes: a new geological estimate of argon blocking temperature for biotite, *EOS, Trans. Am. Geophys. Union*, **57**(4), 353, 1976.
- UENO, H., E. IRVING, and R.H. McNUTT, Paleomagnetism of the Whitestone anorthosite and diorite, the Grenville polar track, and relative motions of the Laurentian and Baltic shields, *Can. J. Earth Sci.*, **12**, 209–226, 1975.
- VITORELLO, I. and R. VAN DER VOO, Late Hadrynian and Helikian pole positions from the Spokane Formation, Montana, *Can. J. Earth Sci.*, **14**, 67–73, 1977.
- WU, Y.T., M. FULLER, and V.A. SCHMIDT, Microanalysis of N.R.M. in a granodiorite intrusion, *Earth Planet. Sci. Lett.*, **23**, 275–285, 1974.
- YORK, D., A formula describing rock magnetic and isotopic blocking temperatures, *Earth Planet. Sci. Lett.*, 1977a (in press).
- YORK, D., Magnetic Blocking Temperatures, *Earth Planet. Sci. Lett.*, 1977b (in press).
- ZIJDERVELD, J.D.A., A.C. demagnetization of rocks: Analysis of results. In *Methods in Paleomagnetism*, edited by D.W. Collinson, K.M. Creer, and S.K. Runcorn, p. 254–286, Elsevier, Amsterdam, 1967.