Thermal history of the 3.5–3.2 Ga Onverwacht and Fig Tree Groups, Barberton greenstone belt, South Africa, inferred by Raman microspectroscopy of carbonaceous material

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ABSTRACT

Raman spectra of carbonaceous material were collected in situ from samples of cherts of the Onverwacht and Fig Tree Groups in the central Barberton greenstone belt. The spectra feature two dominant peaks characteristic of disordered carbon: the D peak at ∼1310 cm^{-1} and the O peak at 1580–1600 cm^{-1}. D peak positions and relative peak intensities and areas indicate that all samples have been altered to lower greenschist facies or above. No correlation was observed between maximum temperature and stratigraphic position or degree of hydrothermal alteration, implying that metamorphism in the central Barberton greenstone belt was regional and unaccompanied by the flow of large quantities of hydrothermal fluids. Samples from the Marble Bar Chert of the Pilbara block, Western Australia, have been heated to the same extent as samples from Barberton. This study demonstrates the use of Raman spectra of carbonaceous material as a sensitive geothermometer for low-temperature metamorphic facies. This application could also be used to establish the antiquity of putative microfossils from metamorphic terranes.

Keywords: carbonaceous rocks, chert, metamorphism, Barberton greenstone belt, Raman spectra.

INTRODUCTION

The Swaziland Supergroup of the Barberton greenstone belt, South Africa, is one of the oldest, relatively unmetamorphosed volcanic and sedimentary sequences on Earth. Most rocks in the belt have been subjected to some degree of metasomatism and metamorphism, commonly to greenschist grade, but lack shear fabrics and evidence of penetrative deformation. However, many of the sedimentary rocks are cherts composed largely of silica with trace amounts of carbonaceous material, and they lack metamorphic mineral assemblages that might record their thermal histories. The carbonaceous material in these rocks has been interpreted as biological in origin and may contain the oldest recognized microfossils (Walsh and Lowe, 1985, 1999). The lack of a conventional high-resolution geothermometer for many Barberton rocks makes interpretation of both the degree of alteration of carbonaceous material and the metamorphic history of the greenstone belt problematic.

Several studies have demonstrated the potential as a geothermometer of laser Raman microspectrometry of partially graphitized carbonaceous material (Beyssac et al., 2002; Jehlicka and Bény, 1992; Roberts et al., 1995; Spötl et al., 1998; Wopenka and Pasteris, 1993; Yui et al., 1996). Once carbonaceous material has reached approximately greenschist facies, it undergoes a characteristic loss of noncarbon atoms (e.g., hydrogen) and conversion to increasingly large graphite crystallites, both reflected by its Raman spectra (Grew, 1974; Wopenka and Pasteris, 1993). In this study, laser Raman spectroscopy has been applied as a geothermometer to samples of carbonaceous chert from the Onverwacht and Fig Tree Groups (Fig. 1). Because of the abundance of carbonaceous cherts in the Onverwacht Group (Lowe, 1999) and the general lack of diagnostic metamorphic mineral assemblages in many sedimentary units, this geothermometer could be a useful tool for constraining the thermal history of these and similar rocks.

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GEOLOGIC SETTING

The stratigraphy of the Barberton greenstone belt was summarized by Lowe and Byerly (1999). The Swaziland Supergroup is divided into the basal, predominantly volcanic Onverwacht Group and the succeeding sedimentary Fig Tree and Moodies Groups (Fig. 1). The two lowest units of the Onverwacht Group, the Sandspruit and Theespruit Formations (Viljoen and Viljoen, 1969), are in fault contact with the rest of the group. Of the two, only the Theespruit contains significant sedimentary units.

The other four formations of the Onverwacht Group (Komati, Hooggenoeg, Kromberg, and Mendon Formations) and the Fig Tree Group form a continuous stratigraphic sequence. The Komati Formation is a thick (∼3.5 km) accumulation of komatiitic volcanic rocks with no major sedimentary units. The Hooggenoeg Formation (∼3.8 km) consists of cycles of komatiitic and basaltic flows capped by thin sedimentary units. The overlying Kromberg Formation includes ∼2.3 km of mostly mafic volcanic and volcaniclastic rocks, but the lowest 150–350 m is a unit of silicified carbonaceous and ferruginous sediment called the Buck Reef Chert. The Mendon Formation (∼1 km) is composed of cycles of komatiitic volcanic rocks capped by thin cherty sedimentary units.

METHODS

The studied samples are carbonaceous black or black-and-white banded cherts. All but two were collected away from known intrusive bodies. One sample was collected from the Buck Reef Chert within the 1–3-km-wide contact aureole around the 3216 Ma Dalmain pluton, a large body of granitoid rock. Another sample is a carbonaceous chert xenolith from within a basaltic dike in the Buck Reef Chert. Two samples from the Fig Tree Group came from mines in areas of high strain and gold mineralization along the northern edge of the belt. Five samples are clasts of carbonaceous chert from conglomerates of the Moodies Group. Also, two samples from the Marble Bar Chert of the Pilbara block, Western Australia, were analyzed. No samples showed evidence of strain except for those from the mines. Nearly every chert unit was sampled at several different localities. As is typical for Barberton carbonaceous cherts (Walsh and Lowe, 1999), carbonaceous material observed in thin sections from the studied samples is generally present as disseminated grains between 5 and 1000 μm in diameter completely surrounded by chert.

Raman spectra were collected in situ from unpolished slabs of chert to avoid polishing effects (Pasteris, 1989). The instrument used was a Kaiser Hololab D5000 Raman microscope equipped with a 785 nm diode laser oriented normal to the sample. This instrument had a spot size of ∼1 μm, an effective 4000

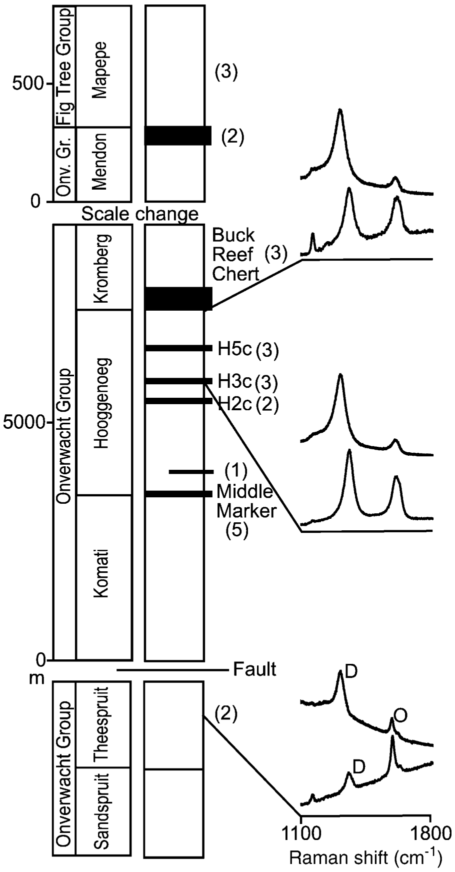

Figure 1. Schematic section of Onverwacht and Fig Tree Groups emphasizing sedimentary units sampled in this study (modified from Lowe and Byerly, 1999). Numbers in parentheses indicate number of sampling localities for each unit. Representative Raman spectra from several units are shown. In each case, bottom spectrum was observed with excitation wavelength of 514.5 nm; top spectrum was observed at 785 nm. D and O peaks are labeled on lowest spectra.

channels, and 4 cm^{-1} resolution. An average power of $\sim$40 mW was applied at the sample surface. These spectra were compared to spectra collected at low laser power ($\sim$1 mW) to test for sample damage; no permanent changes in spectra were observed due to laser-induced heating.[1] Raman spectra were collected from individual carbonaceous grains identified as discrete dark regions under high magnification for 100 s or longer. Spectral features were interpreted by comparison with known reference materials including graphite, kerogens, quartz, and carbonates. Features attributed to mineral phases were not analyzed further. Because spectral features used to infer degree of crystallinity of disordered carbonaceous material vary depending on the orientation of crystallites to the exciting laser beam, single measurements from unoriented samples can be used to infer minimum degrees of heating (Pasteris, 1989). To obtain estimates of the actual degree of heating, spectra were collected from 2–10 different points in each sample. Care was taken to obtain many spectra from samples inferred to be the least heated representatives of individual units.

To quantify peak parameters, spectra were fitted with mixed Gaussian-Lorentzian curves. The broad shoulder at the low-wave-number side of the D peak was approximated as the sum of a broad peak at $\sim$1220 cm^{-1}, a weaker narrow peak at $\sim$1160 cm^{-1}, and a weaker broad peak at $\sim$1070 cm^{-1}. No attempt was made to interpret these minor peaks; this template was found to give stable, reproducible fits. The D peak position as quantified by the position of the major fitted peak in the 1310 cm^{-1} region was found to be the best indicator of metamorphic grade, so the point yielding the highest-energy position was chosen for analysis. The D peak position shifted up $\sim$2.5 cm^{-1} during measurement at 40 mW as compared to 1 mW. This shift was constant with respect to the absolute position measured at 40 mW. Since all data used for estimation of metamorphic grade were collected at 40 mW, this shift does not affect position variations or grade assignments.

For comparison to the geothermometer developed by others using argon ion lasers (514.5 nm), spectra were collected at 2 to 4 points from each of 11 samples using a Renishaw M1000 MicroRaman spectrometer equipped with an argon ion laser at the California Institute of Technology. An average power of $\sim$0.5 mW was applied at the sample surface. Data collection proceeded as previously described.

RESULTS

Two peaks characteristic of disordered carbonaceous material were observed in the studied samples (Fig. 1). The O peak is located at $\sim$1580–1600 cm^{-1} and results from in-plane vibrations in ordered graphite. In polycrystalline graphite, a shoulder appears at $\sim$1610–1620 cm^{-1}. In very poorly ordered carbonaceous material, this shoulder is not resolved from the O peak, which appears as one broad peak at $\sim$1600 cm^{-1}. The D peak results from a resonant Raman process in poorly ordered carbonaceous material. Observed with a 514.5 nm laser, the D peak appears at $\sim$1350 cm^{-1}. With a 785 nm laser, it appears at $\sim$1310 cm^{-1} (Matthews et al., 1999).

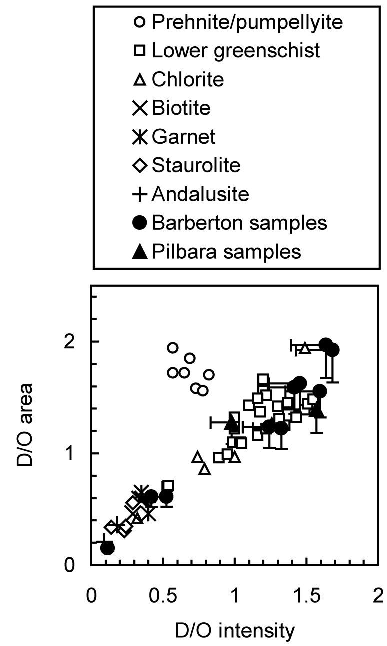

Figure 2. Plot of D/O area vs. D/O intensity discriminates between low-grade samples. Prehnite-pumpellyite facies and lower greenschist facies data are from Yui et al. (1996). Chlorite zone through andalusite zone data are from Wopenka and Pasteris (1993). Barberton and Pilbara samples are from current study. Error bars are –15%, standard deviation for each ratio for multiple grains from same sample.

Previous studies have shown that D/O height, width, and area ratios determined with a 514.5 nm laser vary systematically with increasing metamorphic grade in metapelites (Beyssac et al., 2002; Jehlicka and Bény, 1992; Roberts et al., 1995; Spötl et al., 1998; Wopenka and Pasteris, 1993; Yui et al., 1996). Data from studies that measured these ratios in similar ways were compiled in discriminant charts for comparison with the calibration spectra from this study (Fig. 2). This comparison suggests that the least metamorphosed samples examined in this study have carbonaceous material with crystallinity equivalent to that in chlorite-zone shales representing the lower greenschist facies. Since development of coarse crystallinity in carbonaceous material is enhanced in cherts and carbonates (Grew, 1974; Pasteris and Chou, 1998; Wintsch et al., 1981), these samples have experienced peak temperatures at most equal to those of chlorite-zone shales. The most metamorphosed samples are at least equivalent to chlorite-biotite–zone shales or just below middle greenschist facies. The two samples from the Marble Bar Chert of the Pilbara block appear to record temperatures comparable to those recorded in the Barberton greenstone belt. This result is consistent with previous as-

[1]GSA Data Repository item 2004004, a table of D peak positions for each sample included in this study and a figure showing spectra collected to test for heating effects, is available online at www.geosociety.org/pubs/ft2004.htm, or on request from editing@geosociety.org or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301-9140, USA.

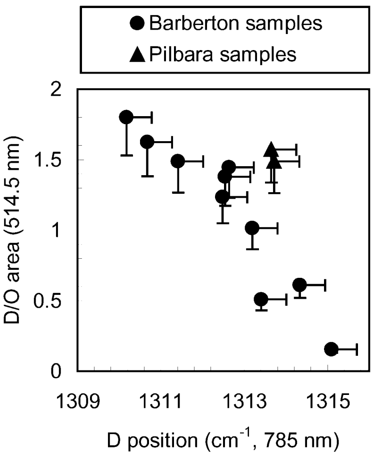

Figure 3. D position measured with 786 nm laser correlates with D/O area ratio measured with 514.5 nm laser. D positions plotted are maximum values observed for several (about five) grains from same sample. Area ratios are minimum values observed for about four grains from each sample and predict minimum degree of heating that affected associated sample. Error bars shown for D position are +0.6 cm^{-1}, about reproducibility of fit. Error bars shown for area ratio are −15%, standard deviation of that ratio for multiple grains from same sample.

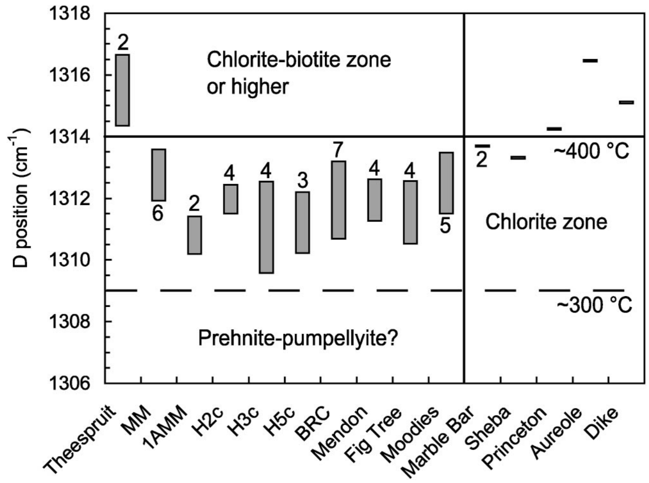

Figure 4. Range of D positions observed in samples by unit. Placement of prehnite-pumpellyite line is uncertain. Temperature is unlikely to be linear in this scale. MM—Middle Marker; 1AMM—First chert above Middle Marker; BRC—Buck Reef Chert; other units as in Figure 1. Mine samples from Fig Tree Group are labeled Sheba and Princeton after mines from which they were collected. Number of samples analyzed from each unit is noted above or below bar. If no number is indicated, then only one sample was analyzed.

signments of lower greenschist facies to the rocks of the Marble Bar belt (Hickman, 1983).

The D peak position measured with the 785 nm laser was highly correlated to metamorphic grade as determined from D/O area ratio measured with the 514.5 nm laser, although the Pilbara samples seem to fall off the line described by the Barberton samples (Fig. 3). A weak correlation between thermal maturity and D peak position measured with 514.5 nm lasers has been previously observed (Spötl et al., 1998), but such a correlation is the exception rather than the rule. There is no known mechanism to explain the correlation observed here.

This correlation was exploited to assign a metamorphic grade to all Barberton samples studied, although the somewhat different behavior of the Pilbara samples suggests that this correlation might not apply without modification to other units. Figure 4 shows the ranges of D peak positions measured for samples in each stratigraphic unit (see footnote 1). Significant variation is evident within the Onverwacht Group as a whole and within individual units. The samples that record the highest temperatures are severely recrystallized chert from the Theespruit Formation. Except for H2c, the lowest temperature samples from every unit above the Middle Marker record temperatures having distributions that can be explained by an ~0.6 cm^{-1} reproducibility in peak position fit, i.e., that are nearly indistinguishable. Carbonaceous material from H2c and part of the Buck Reef Chert may have developed greater crystallinity because of higher amounts of trace carbonate in those units rather than because of exposure to higher peak temperatures.

The two samples from contact-metamorphic zones show two of the highest apparent temperatures in the sample suite. Two of the Fig Tree Group samples collected in mines also show higher-than-average apparent temperatures. This result is not surprising given the extensive local shearing and mineralization that have affected rocks in this area.

DISCUSSION

Raman microspectroscopy of carbonaceous material in the Onverwacht and Fig Tree Groups in the Barberton greenstone belt indicates a relatively low degree of metamorphism. In particular, the stratigraphically continuous Hooggenoeg, Kromberg, and Mendon Formations of the Onverwacht Group and the overlying Fig Tree Group have all been subjected to temperatures at most equal to those reflected in chlorite-zone shales, i.e., between ~300–400 °C (Bucher and Frey, 1994). These results are in agreement with mineral assemblages observed in Onverwacht volcanic units (Viljoen and Viljoen, 1969). De Ronde et al. (1991) and Toulkeridis et al. (1998) estimated regional heating to a minimum of 200 °C on the basis of isotopic resetting in barites and carbonates, respectively, in agreement with the results presented here. Xie et al. (1997) applied a chlorite geothermometer to estimate an ~320 °C temperature of metamorphism for volcanic rocks in the Onverwacht Group. The latter estimate is close to the onset for chlorite-zone shale metamorphism, suggesting that the maximum temperatures inferred for Barberton cherts by comparison to shales may be very close to the actual temperatures.

The fact that most variation in metamorphic grade above the base of the Hooggenoeg Formation occurs within rather than between units may suggest that most of the variation seen above the regional metamorphic background is due to variations in proximity to dikes, sills, or other shallow intrusions or to other regions of high-temperature alteration. It is also possible that some variation may be due to differences in primary carbonaceous material (Kribek et al., 1994) or to heterogeneities within grains (Buseck and Huang, 1985).

Knauth and Lowe (2003) measured oxygen isotope compositions of cherts from throughout the Barberton greenstone belt and found that cherts below member H6 of the Hooggenoeg Formation have been isotopically reequilibrated with surrounding volcanic rocks. They interpreted this resetting to be the result of water-rich hydrothermal alteration associated with a magmatic event at 3445 Ma and not simply metamorphism at a higher temperature. Stratigraphically higher units show nonequilibrated compositions and have not been similarly altered. Because the Raman spectra of units below H6 do not generally record temperatures higher than those exhibited by overlying units, the present results support the interpretation that thermal metamorphism alone was not a significant factor in determining the oxygen isotope composition of cherts in the belt. Also, although there was also extensive alteration of units above H6 (e.g., Toulkeridis et al., 1998), water:rock ratios were insufficient to reset $\delta^{18}O$ values in units otherwise heated to comparable metamorphic temperatures.

Oxygen isotope analysis indicates that the Moodies chert clasts were derived from below

H6 of the Hooggenoeg Formation (Knauth and Lowe, 2003). Raman microspectroscopy shows that these clasts were heated to temperatures at least as high as the source units. Either the lower members of the Hooggenoeg Formation had already achieved their maximum temperature when the Moodies Group was deposited, which seems unlikely, or heating of all groups was later, regional, and uniform across the central Barberton greenstone belt.

CONCLUSIONS

Raman spectra of carbonaceous material from 47 samples of cherts representing nearly every major sedimentary unit of the Onverwacht Group were used to evaluate the metamorphic grade and peak metamorphic temperatures that have affected rocks in the Barberton greenstone belt. When measured with a 785 nm laser, D peak position correlated with metamorphic grade inferred by spectral characteristics measured with a 514.5 nm laser on a subset of samples. This correlation was used to estimate metamorphic grade of every sample. With the exceptions of the Theespruit Formation, the Middle Marker, and H2c, the least heated samples from every unit of the Onverwacht Group have been heated to nearly the same degree. This result suggests that regional rather than burial or contact metamorphism was responsible for overall background heating in the Onverwacht Group. Maximum heating therefore occurred after deformation of the belt, i.e., after 3.2 Ga (Lowe et al., 1999). This late timing is consistent with indications that Barberton rocks were subjected to temperatures of at least 200 °C as late as 2.7 Ga (de Ronde et al., 1991; Toulkeridis et al., 1998).There is no detectable shift in thermal grade across the major oxygen isotope boundary reported by Knauth and Lowe (2003). This finding confirms that a high flux of hydrothermal fluids was responsible for homogenizing $\delta^{18}O$ of rocks in the Hooggenoeg Formation. The lack of oxygen isotopic resetting in cherts above the Hooggenoeg Formation, despite equal degrees of heating, indicates that regional hydrothermal activity in the greenstone belt was never as significant as during the emplacement of the 3445 Ma magmatic suite.

Raman spectra could be used to test the antiquity of putative microfossils in rocks of comparably heated terranes. Such spectra must be collected from specimens observed below transparent grains in polished thin section to avoid polishing effects (Pasteris, 1989). Any ''microfossil'' yielding a Raman spectrum without well-developed D and O peaks or displaying peaks corresponding to functional groups of thermally unstable organic compounds is a recent contaminant. Conversely, a Raman spectrum indicating metamorphism comparable to that of the surrounding rocks indicates that the carbonaceous material has been in place since the time of maximum heating. In the case of rocks from the Onverwacht and Fig Tree Groups, this is at least 2.7 Ga (de Ronde et al., 1991; Toulkeridis et al., 1998).

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