A COMPARISON OF QUANTITATIVE TECHNIQUES FOR DETERMINING THE 3D ANISOTROPY OF SHALE SAMPLES: APPLICATION TO HORN RIVER BASIN SHALES, BRITISH COLUMBIA, CANADA

Vivian Ebufegha and David K. Potter

Department of Physics, and Department of Earth and Atmospheric Sciences, University of Alberta, Edmonton, Canada

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ABSTRACT

With increasing interest in shale gas and oil plays, an understanding of the anisotropic properties of shales is becoming important for modelling their potential response to stress and their 3D petrophysical properties (such as permeability anisotropy), and thus for exploiting them via hydraulic fracturing. Various studies have mentioned that shales are anisotropic, but a quantitative 3D comparison of different anisotropy parameters is lacking, and details of the factors controlling anisotropy are limited. Part of the reason for this is the fissile nature of many shales, which makes it difficult to cut consolidated core plugs or obtain plugs that remain intact whilst undertaking the anisotropic measurements. We therefore first describe a sample preparation technique which maintains the integrity of shale samples and allows anisotropic measurements (primarily magnetic, but also potentially electrical and acoustic) to be taken on cylindrical or cubic core samples. We then detail quantitative 3D anisotropy measurements on several shale samples from the Horn River Basin in Canada (NW British Columbia, primarily in the Muskwa, Otter Park and Evie formations) by comparing the anisotropy of magnetic susceptibility (AMS), the anisotropy of magnetic remanence (AMR), the anisotropy of electrical resistance (AER), and the observed petrofabric from thin section and polished section analyses. A key result is that several samples exhibit "inverse AMS fabrics" where the maximum magnetic susceptibility axis is perpendicular to the bedding plane and not within it as one would normally expect. This can be explained by the presence of uniaxial stable single-domain (SSD) ferrimagnetic particles, which have a maximum magnetic susceptibility perpendicular to their long axes. Larger multidomain (MD) ferrimagnetic particles, in contrast, have a maximum magnetic susceptibility parallel to their long axes. This effect has been documented in igneous rocks (Potter and Stephenson, 1988), but never before in shales. We demonstrate, therefore, the importance of undertaking AMR measurements (rarely performed on shales before), because the maximum remanence of SSD or MD particles is always along their long axes so the maximum AMR axis will represent the true particle alignment axis. Studies that merely use the more conventional AMS technique could incorrectly infer a preferred particle alignment axis that is 90 degrees

from the true orientation if a significant proportion of SSD particles are present. This may have important consequences for optimizing hydraulic fracturing procedures.

Our results are helping us distinguish different elements of shale anisotropy due to: mineralogy, mineral alignment, organic matter etc. Integration of AMS, AMR, AER, and thin section and polished section analysis suggest that pyrite is a significant control on anisotropy in the Otter Park formation, whereas organic matter appears to be responsible for the high anisotropy in the Evie formation.

INTRODUCTION

Several works have mentioned that shales are anisotropic (Hirt et al, 1995; Lonardelli et al, 2007), but details on the factors that control anisotropy are limited and a comparison of different anisotropy parameters is lacking. Anisotropy in shales is potentially important for optimizing hydraulic fracturing procedures. The primary goal of this study is to determine the degree and orientation of anisotropy in shales of the Horn River Basin by comparing the anisotropies of magnetic susceptibility, magnetic remanence, and electrical resistance, along with oriented thin sections and polished sections. These techniques helped in the assessment of the relationship between elements of shale petrofabric, such as mineralogy, preferred mineral orientation, and organic matter. Subsurface core from three wells was used for this study. Most of the measurements were undertaken from one well, which consisted of a 200 m cored section that penetrated all of the Muskwa, Otter Park and Evie formations.

SAMPLE PREPARATION

Considerable portions of the Horn River formations are extremely friable, and hence did not enable us to cut standard cylindrical core plug samples in many intervals. It was therefore necessary to develop a technique that ensured the samples remained intact during cutting. It was important that the adhesive chosen be fluid enough to sufficiently penetrate the sample. It was also necessary that its magnetic properties not mask those due to the rock fabric. Some paleomagnetic studies have reported coating poorly consolidated samples with weakly diamagnetic sodium silicate (Na_2SiO_3) prior to taking measurements (Hall and Sager, 1990). The fissile nature of the samples used in the present study required that they be impregnated with the adhesive by soaking for 3-5 minutes and then dried for 1-2 days before cutting. Initial attempts to cut samples under water proved futile since sodium silicate is water soluble. Cutting the samples under a jet of propylene glycol, however, ensured the adhesive holding the samples together remained intact. This method ensured minimal alteration of the original fabric prior to the taking of measurements. For the sodium silicate impregnated shales cubic samples with dimensions 2.2cm×2.2cm×2.2cm were cut. It was possible to cut cylindrical plugs from some sufficiently consolidated core sections. These plugs were 2.2cm diameter and 2.2cm long (without sodium silicate impregnation) from sections of the Muskwa and Otter Park formations, and were used for the comparisons of anisotropy of magnetic susceptibility (AMS) and anisotropy of electrical resistance (AER).

ANISOTROPY METHODS

Thin section and polished section petrofabric analysis

Thin section petrography is commonly used to visually characterize anisotropy (Chalmers et al, 2012). Whilst this method is limited by its dependence on the presence of visible laminations and the preferred orientation of large enough minerals, it provides a means of explaining the results of other techniques like magnetic anisotropy. The minerals or fabric elements controlling anisotropy may also be confirmed with thin sections. The petrofabric analysis was supplemented with polished sections and associated reflected light microscopy.

Anisotropy of Magnetic Susceptibility (AMS)

Magnetic susceptibility is a second rank tensor described by an ellipsoid with principal axes $k_1 \geq k_2 \geq k_3$ for an anisotropic sample, where k_1 is the maximum susceptibility axis and k_3 is the minimum susceptibility axis. For any given sample, the susceptibility ellipsoid represents the average preferred orientation of grains or the average grain shape. For a random (isotropic) arrangement of grains, the ellipsoid is spherical ($k_1=k_2=k_3$). Anisotropy of magnetic susceptibility can arise due to the preferred orientation of the mineral grains. With increasing degree of preferred orientation, such as might be induced by compaction in shales, one might expect the AMS ellipsoid to become more oblate (disk shaped with $k_1=k_2 > k_3$). This would be regarded as a "normal" magnetic fabric. In such a case the minimum k_3 axis would be perpendicular to bedding and would have a high inclination value (close to 90°) if the bedding is close to horizontal. However, an important property, which is not often taken into consideration, is that AMS is dependent upon the domain state and therefore grain size of any ferro- or ferrimagnetic grains that may be present. Grains larger than about 1μm (depending upon composition, aspect ratio, etc) are called multidomain grains, and have their maximum susceptibility along particle long axes and minimum susceptibility perpendicular to particle long axes (Stephenson et al, 1986). In contrast, uniaxial stable single domain grains (generally submicron) have their maximum susceptibility direction perpendicular to the long axis of the grain while the minimum susceptibility is along the long axis of the grain. A predominance of uniaxial stable single domain grains can give rise to so-called "inverse" magnetic fabrics, where the minimum k_3 axis is parallel to bedding and has a low inclination value (close to 0°) if the bedding is close to horizontal. No previous studies have shown such behaviour in shales, but we will demonstrate the behaviour in several samples in the present study.

In the present study we used a Bartington (MS2B) static AMS meter. The static AMS meter is unidirectional, measuring susceptibility in one direction for each application of a low field of 80 Am^{-1} to a sample of known mass in a $10cm^3$ plastic slot. The instrument measures the magnetization induced in the sample by the applied magnetic field and calculates the ratio between both the induced magnetization and the applied field (magnetic susceptibility) (Dearing, 1999). The AMS meter requires that samples be turned manually in a desired number of directions to obtain directional susceptibility. In this study, magnetic susceptibility using the static AMS meter was undertaken in 18 different directions for each sample. The corresponding magnetic susceptibility ellipsoid was then computed. Each directional measurement of magnetic susceptibility takes between one and two minutes. This measurement scheme was chosen so as to compare directly with the same scheme involving 18 measurements of anisotropy of electrical resistance (AER) as described below.

Anisotropy of Magnetic Remanence (AMR)

Remanent magnetization is magnetization retained by minerals after the removal of an applied field. It is a characteristic of ferro- or ferrimagnetic minerals (e.g., magnetite). Like magnetic susceptibility, magnetic remanence can be described by an ellipsoid with three orthogonal principal axes. The measurements can either be made by applying a magnetic field to generate a magnetic remanence along the same 18 different directions in the sample as described for the AMS measurements, or by applications of an applied field along 3 orthogonal reference axes, which generates 9 components of magnetization (Stephenson et al, 1986). Tumble alternating field (AF) demagnetization is employed prior to each new remanence acquisition step. Anisotropy of laboratory induced isothermal remanent magnetization (AIRM) was used in this study. Isothermal remanent magnetization is the remanence acquired in a direct field at constant temperature, and was imparted using a pulse magnetizer in the present study. The pulse lasts for about 100 ms. The method is relatively quick and gives the highest signal for any type of magnetic remanence (and therefore is useful for samples with low ferrimagnetic mineral content). Unlike magnetic susceptibility, the maximum and minimum directions of magnetic remanence are not dependent on magnetic domain state. For both multidomain and stable single-domain grains the maximum remanence is along the long axis of the grain and the minimum perpendicular to the long axis (Stephenson et al, 1986).

Anisotropy of Electrical Resistance (AER)

Measurements of the anisotropy of electrical resistance (AER) were made on consolidated cylindrical core plugs from the Muskwa and Otter Park formations. These plugs were not impregnated with sodium silicate as we wanted to ensure that the AER measurements would not be affected by the impregnation process. Slight changes in the original micro fracture distribution that might result from the sodium silicate impregnation might possibly have a small effect on AER measurements, but should have virtually no effect on AMS measurements. In this study AER is described by a second rank tensor computed from the electrical resistances measured in multiple directions. The electrical resistance tensor can, therefore, be easily compared with those of other properties such as the magnetic susceptibility tensors. A Keithley 614 electrometer was used in the present study, which measures electrical resistances up to 200 giga ohms. The sample is fixed between two copper plates held in place by a plastic clamp and connected to the electrometer by a positive and negative electrode. The copper plates ensure good electrical contact between the electrodes and the rock surface. A current of 100×10^{-12} A is passed from the electrometer through the positive electrode, sample and negative electrode. The directional resistance is calculated in the circuit, and is a measure of the resistance to the flow of current in that direction. To minimize uncertainty, an average of resistances over a thirty minute interval was taken for each sample direction. The AER measurements were directly compared with AMS measurements on identical cylindrical core plug samples using exactly the same Bartington scheme of 18 different directional measurements per sample.

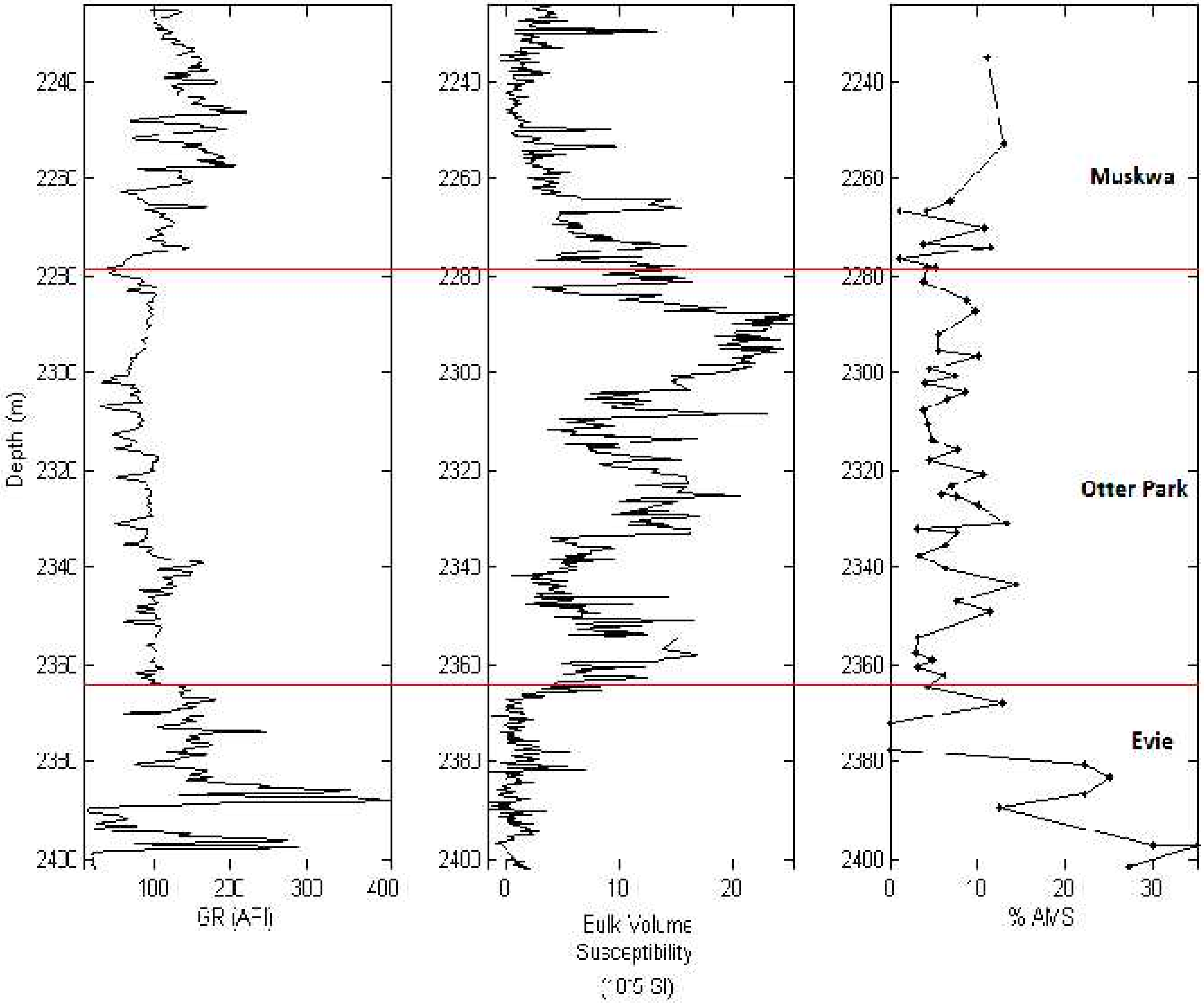

Figure 1. (Left) Wireline gamma ray, (middle) bulk volume magnetic susceptibility and (right) percentage anisotropy of magnetic susceptibility (AMS) with depth through a 200 m section of the Muskwa, Otter Park and Evie formations of the Horn River shales. AMS is highest in the Evie (most likely due to organic matter) despite its low bulk susceptibility. AMS varies in the Otter Park due to variations in pyrite content.

RESULTS AND DISCUSSION

Magnetic anisotropy (AMS and AMR) integrated with observed petrofabric

Figure 1 (left) shows the wireline gamma ray through the studied interval, and Figure 1 (middle) shows our measured bulk volume magnetic susceptibility of slabbed core using a Bartington MS2E probe sensor. The latter measurements were taken every 0.25m. In a previous study of shoreface reservoirs Potter et al (2004) showed that where the magnetic susceptibility is controlled by paramagnetic clays, especially illite, the magnetic susceptibility and paramagnetic clay content correlate with the natural gamma ray signal. Figure 1 (left and middle) shows that this is not the case in the Horn River shales. The absence of a correlation between bulk volume susceptibility and gamma ray emission suggests that the observed variations in susceptibility may not merely be the result of varying clay content.

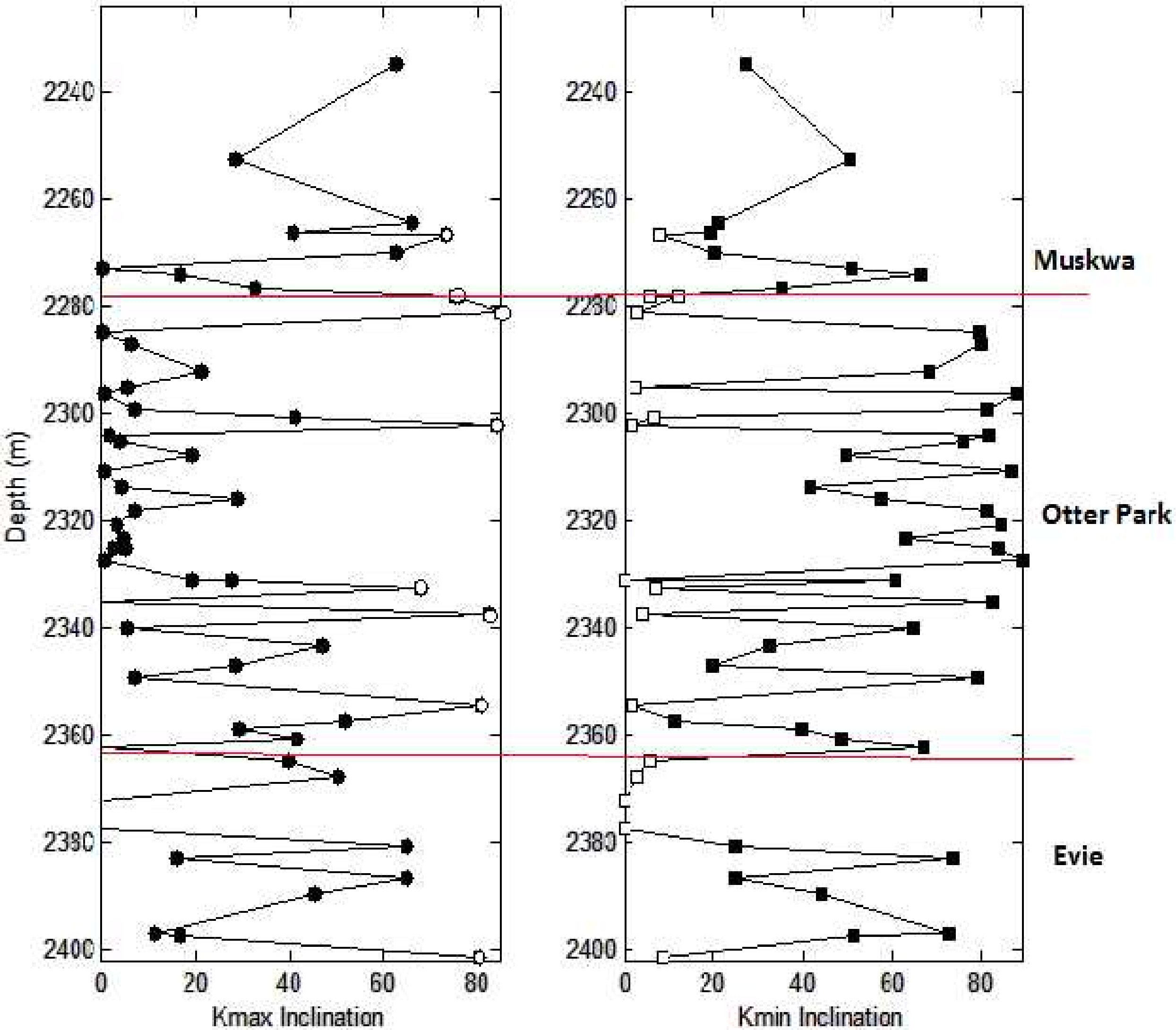

Figure 2. Variation of inclination (in degrees) of maximum (kmax) and minimum (kmin) susceptibility principal axes with depth for a 200 m section through the Muskwa, Otter Park and Evie formations of the Horn River shales. Closed symbols indicate "normal" fabrics with kmax close to horizontal (low inclination) and kmin close to vertical (high inclination), and open symbols indicate "inverse" fabrics with kmax close to vertical (high inclination) and kmin close to horizontal (low inclination).

Anisotropy of magnetic susceptibility (AMS) ellipsoids for 57 cubic shale samples impregnated with sodium silicate, using the Bartington scheme of measurements in 18 different orientations per sample (by rotating the sample in a holder), showed that the shales in the Muskwa, Otter Park and Evie formations are all magnetically anisotropic**.** Figure 1 (right) shows the percentage AMS with depth, where percentage AMS is given by 100 $(k_{max}–k_{min})/k_{int}$. Hirt et al (1995) showed that AMS increased with depth in Devonian shales due to the compaction induced alignment of minerals. In the present study, however, the percent AMS in the Muskwa and the Otter Park is very variable, and is only significantly higher in part of the Evie. The controls on anisotropy vary in each formation. For the 57 samples in Figure 1 (right), the inclinations of the k_{max} and k_{min} AMS axes are shown in Figure 2. The results surprisingly show an alternation of normal and inverse AMS fabrics. Inverse AMS fabrics have not been reported for shales before, but have been documented for igneous rocks (Potter and Stephenson, 1988). They can arise from ferrimagnetic uniaxial stable single domain particles (such as magnetite) as described in the Anisotropy Methods section. To confirm whether this was the reason here we undertook AMR measurements. This involved giving the samples an isothermal remanent magnetization (IRM) using a pulse magnetizer. Due to the size and shape of the samples in relation to the pulse magnetizer sample holder we were unable at this stage to undertake full AMR ellipsoids by applying the direct field (DF) in all sample orientations. However we were able to undertake IRM measurements parallel and perpendicular to the bedding. For all samples the IRM parallel to bedding was higher than that perpendicular, irrespective of whether the AMS fabric was normal or inverse. This strongly suggests that the inverse AMS fabrics are due to the presence of uniaxial stable single domain particles. Figure 3 shows thin sections through a sample from the Muskwa formation that exhibited an “inverse” AMS fabric.

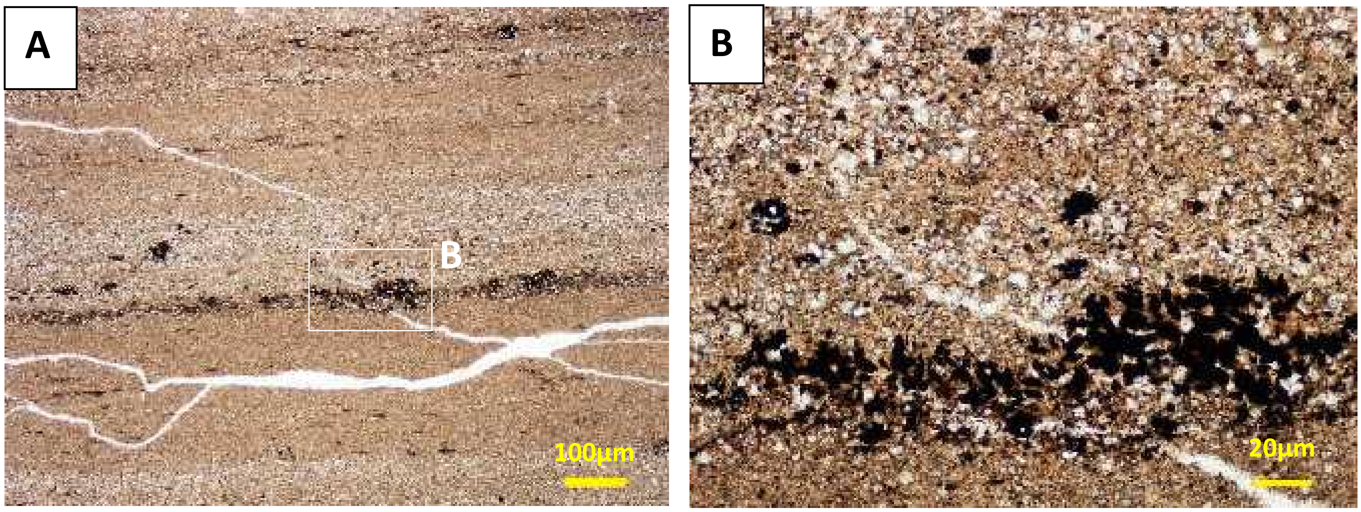

Figure 3. (A) Thin sections perpendicular to bedding through the Muskwa formation at depth 2266.5m. (B) Magnified view of the rectangle in (A). The black layers are clusters of pyrite. The maximum susceptibility axis has an inclination of 73.7º (closely perpendicular to bedding) and the minimum susceptibility has an inclination of 7.9º (closely parallel to bedding). This suggests an “inverse” magnetic susceptibility fabric.

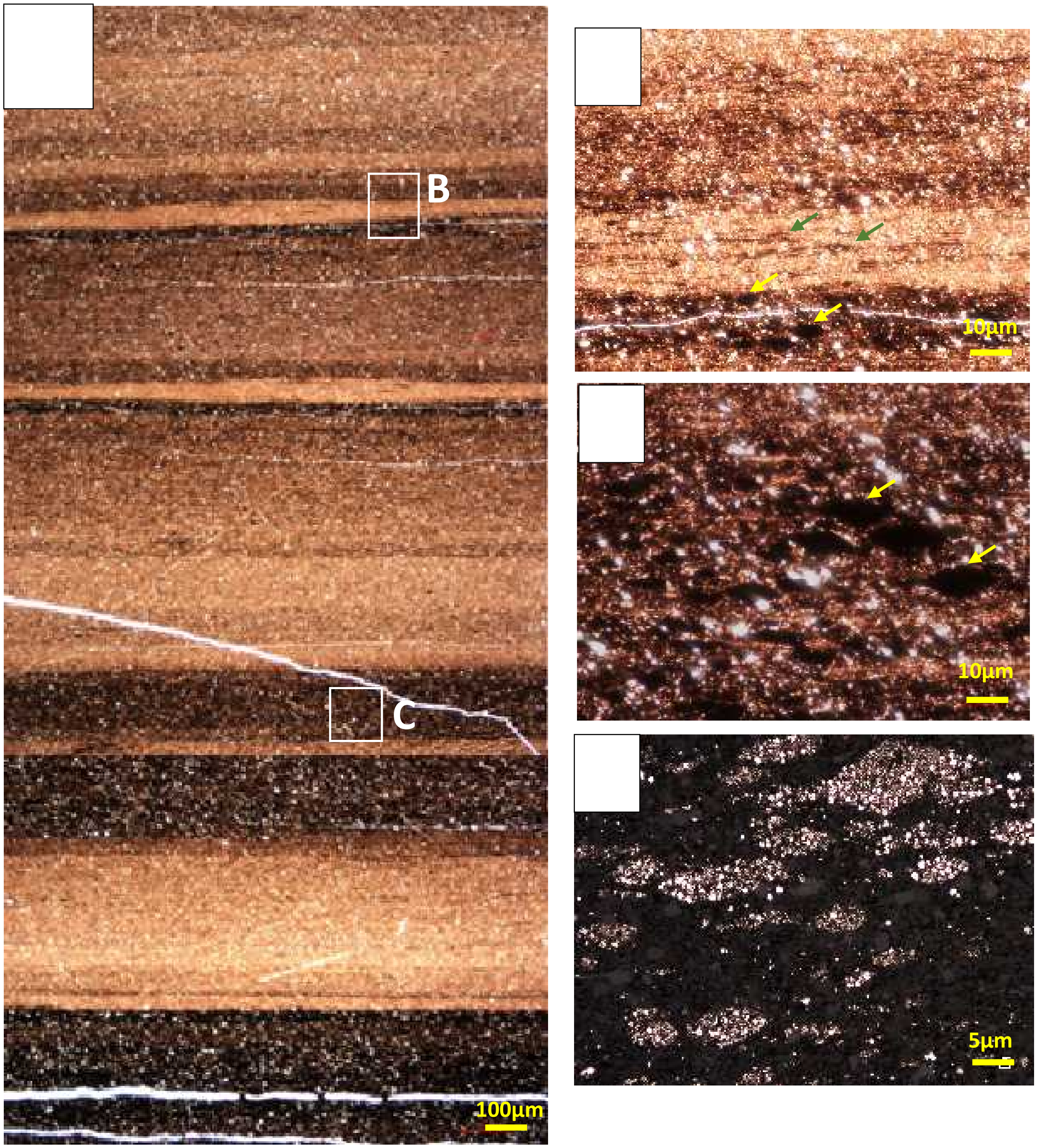

Figure 4. A-C: Thin sections perpendicular to bedding through the Otter Park formation at depth 2274m. B and C are magnified views of the regions indicated in A. Black lenses (yellow arrows in B and C) are elongate, bedding parallel clusters of pyrite. Green arrows in B show strings of organic matter. D: Reflected light image of a polished section of the pyrite lenses showing that the lenses comprise smaller pyrite particles (white areas). The maximum susceptibility axis has a low inclination of 16.7º and the minimum susceptibility axis has a high inclination of 66.1º. This suggests a "normal" magnetic susceptibility fabric.

Thin sections and reflected light images in the three formations help us to interpret the magnetic anisotropy results. The Muskwa formation consists mainly of diamagnetic minerals as shown by the low often negative magnetic susceptibility values in Figure 1 (middle). Cross-polarized light images of the thin sections in Figure 3 show that these are mainly carbonates with minor amounts of quartz (Figure 3 shows the images merely in plane polarized light). Planar lamination is visible in Figure 3A, which represents the bedding plane. The thin dark layers are due to clusters of pyrite grains. This sample at depth 2266.5 m is one that exhibits an inverse AMS fabric, with the minimum susceptibility axis having a very low inclination and being close to the bedding plane. A normal fabric would have the minimum susceptibility inclination close to 90° (perpendicular to the bedding plane). Figure 3B shows a magnified view of an area in Figure 3A. This sample is relatively poor in clay and organic matter, but other samples in the Muskwa have higher clay and organic contents. The white lines in the thin sections are splits induced during the production of the sections (not intrinsic fractures).

Thin sections through the Otter Park formation (Figures 4A-C) show bedding parallel laminations. The Otter Park contains a higher content of paramagnetic minerals compared to the Muskwa as suggested by the higher positive values of magnetic susceptibility in Figure 1 (middle), including pyrite (the dark layers in Figure 4). The pyrite rich layers consist of elongate lenses of pyrite (Figures 4B-D), and grade into more silty layers with strings and lenses of organic matter. Reflected light microscopy (Figure 4D) shows that the lenses of pyrite in Figures 4B-C are made up of smaller grains of pyrite. The pyrite lenses are elongated parallel to bedding and are strongly aligned suggesting that pyrite may be a significant contributor to anisotropy in the Otter Park formation. The strings of organic matter and clay in the less pyritic intervals also show strong bedding parallel alignment. The sample in Figure 4 (at depth 2274 m) exhibits a normal AMS fabric.

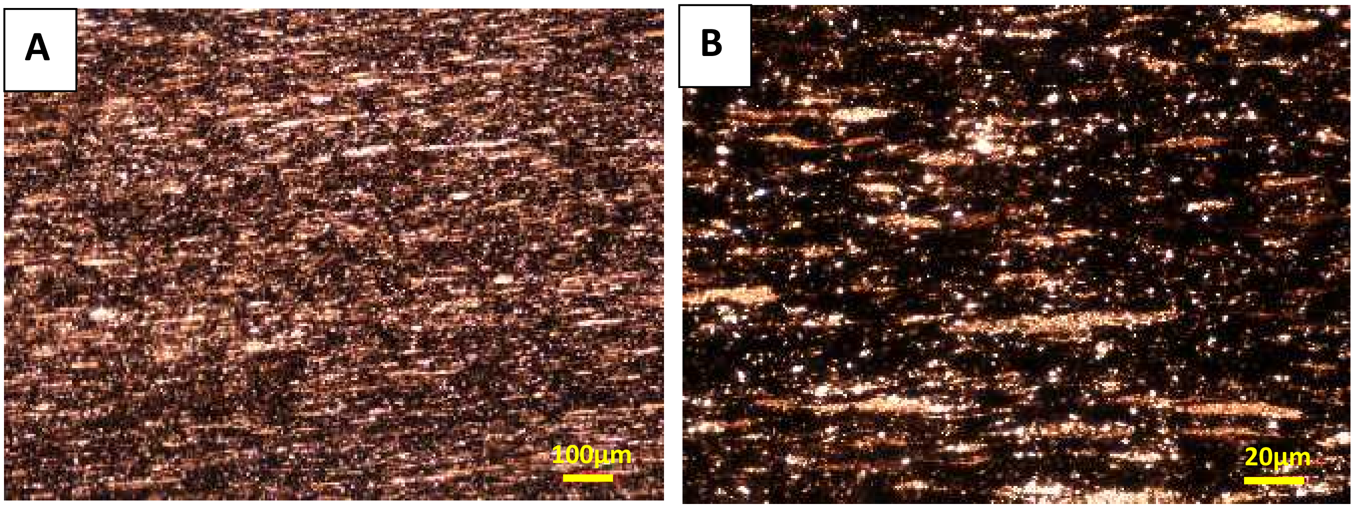

Figure 5. Thin sections perpendicular to bedding through the Evie formation at depth 2367.7m. There are strongly aligned calcite/dolomite clusters in an organic rich matrix. The maximum susceptibility axis has a relatively high inclination of 50.4° and the minimum susceptibility axis has a low inclination of 3.1° (close to horizontal). This suggests an "inverse" magnetic susceptibility fabric.

Thin sections through the Evie formation (Figure 5) show elongate clusters of bedding parallel carbonate grains (dolomite/calcite) in an organic rich matrix. This is consistent with the low and often negative magnetic susceptibility (diamagnetic) values shown in Figure 1 (middle). The distinct bedding parallel preferred orientation of carbonate clusters in Figure 5 suggests that the Evie is quite anisotropic, and is consistent with the higher AMS results in Figure 1 (right). It is unlikely that the large anisotropy could result from the diamagnetic carbonate grains, and so it seems likely that the anisotropy is controlled by the organic rich matrix. The sample in Figure 5 (at depth 2367.7 m) is another that exhibits an inverse AMS fabric.

Anisotropy of Electrical Resistance (AER)

Anisotropy of electrical resistance measurements were undertaken on a separate set of shale samples that were untreated with sodium silicate, where we were able to cut consolidated cylindrical plugs. We avoided at this stage using the cubic sodium silicate treated samples in case the original fracture distribution of these once friable samples may have been altered by the sodium silicate impregnation and may affect the AER results. (Note that the sodium silicate samples would not have had any significant effect on the magnetic anisotropy results, which are influenced by the mineral alignments in the shale). The measurements were undertaken with the samples in their native, dry (not re-saturated) state. We were able to position the samples in the same Bartington scheme of 18 orientations as for the AMS measurements (by moving the sample and/or the copper plates attached to the electrodes). AMS measurements in the same 18 orientations were also undertaken on these samples for direct comparison. Initial results for six samples from the Muskwa and Otter Park formations show that they are all electrically anisotropic (Table 1). The electrical resistance of the Otter Park ($0.1\text{-}0.5 \times 10^9$ ohms) is less than that of the Muskwa ($2\text{-}5 \times 10^9$ ohms), suggesting an increased concentration of conductive minerals like pyrite and clays in the Otter Park.

Table 1 shows that all samples tested have maximum electrical resistance closely perpendicular to bedding (high inclinations) and minimum electrical resistance closely parallel to bedding (low inclinations). This can be explained simply in terms of parallel and series resistors. For a sequence of parallel isotropic layers in a transversely isotropic medium, each layer behaves as a resistor and current applied parallel to the direction of lamination distributes itself through each layer, such that the total resistance (R_{parallel}) is given by $1/R_{\text{parallel}} = 1/R_1 + 1/R_2 + \ldots 1/R_n$. For current flow perpendicular to the direction of lamination, the medium behaves like a set of resistors in series. The same current passes through all layers in the succession, hence the total resistance in the transverse direction is given by $R_{\text{series}} = R_1 + R_2 + \ldots R_n$). For a layered succession with minimal fracture and joint networks, current experiences the least resistance parallel to the direction of grain alignment (Ellis et al., 2010) and maximum resistance transverse to it.

Furthermore, Table 1 shows that the maximum magnetic susceptibility axes show a general correspondence with the minimum electrical resistance axes (low inclinations), and the minimum magnetic susceptibility axes show a general correspondence with the maximum electrical resistance axes (high inclinations). Note that sample 26002-2-10 has a high intermediate magnetic susceptibility inclination (similarly for sample 20613-1-3(1) to a lesser extent), which is a situation that can theoretically result from mixtures of ferrimagnetic uniaxial stable single domain grains and multidomain grains (Ferre, 2002). This also has not been documented in shales before, and again demonstrates the importance of not using AMS alone to infer mineral alignment directions.

Table 1. Inclinations (in degrees) of the AMS principal axes (Kmax, Kint and Kmin) and the AER principal axes (ERmax, ERint and ERmin) for cylindrical plug samples from the Muskwa and Otter Park formations. The maximum ER inclination is high (closely perpendicular to bedding), and the minimum ER inclination is low (closely parallel to bedding). Maximum AMS inclination for most samples also corresponds to minimum AER inclination and vice versa. Note sample 26002-2-10 has a high intermediate magnetic susceptibility inclination (and sample 20613-1-3(1) to a lesser extent), which can result from mixtures of ferrimagnetic uniaxial stable single domain grains and multidomain grains.

Sample	**Kmax Inc**	**ER max Inc**	**Kint Inc**	**ER int Inc**	**Kmin Inc**	**ER min Inc**	**%AMS**
Muskwa							
26002-2-4(1)	19.8	62.1	2.2	1.1	70.1	27.9	8.48
26002-2-7(2)	10.1	74.1	19	6.8	68.3	14.3	28.13
26002-2-10	3.4	69.9	79.5	2.9	9.9	19.8	12.65
Otter Park							
20613-1-3(1)	18	75.5	48.2	0.6	36.2	14.5	19.46
20613-1-6(4)	13.8	62.4	9.4	6	73.2	26.8	10.09
20613-1-9(1)	38.8	68.7	8.5	7.2	49.9	19.9	4.03

CONCLUSIONS

The main conclusions from this work can be summarised as follows:

1. Significant variations were observed in the magnetic anisotropy (AMS and AMR), electrical anisotropy and visual anisotropy (from thin section and reflected light microscopy) of a suite of Horn River shales. The variations are due to differences in mineral content, organic content, and the domain state of the ferrimagnetic grains in the samples. Variations in pyrite content appear to largely control the AMS in the Otter Park, whilst increased organic content in the Evie appears to be responsible for the high values of AMS in this formation.
2. Several samples from the three formations exhibited “inverse” AMS fabrics, where the minimum susceptibility was parallel to bedding rather than perpendicular to bedding in the case of “normal” sedimentary fabrics. Inverse AMS fabrics have been documented previously in igneous rocks, but never before in shales. AMR measurements suggested that the inverse AMS fabrics were due to the presence of uniaxial stable single domain ferrimagnetic grains. Therefore one should not use AMS alone to infer petrofabric (e.g., the bedding orientation) in these shales. Instead AMS should be used in conjunction with

AMR to unambiguously determine the correct petrofabric orientation. Identifying the correct petrofabric orientation will help optimize hydraulic fracturing procedures.

3. Measurements of the anisotropy of electrical resistance showed that all samples studied had maximum electrical resistance near perpendicular and minimum resistance near parallel to the bedding plane. The maximum magnetic susceptibility axes for most of the samples closely corresponded to the minimum electrical resistance axes and vice versa.

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