ELECTRON PARAMAGNETIC RESONANCE (EPR) SPECTROSCOPY AND GEOCHEMISTRY IN TIN EXPLORATION AT RENISON, TASMANIA AUSTRALIA

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Abstract

Rock powder of dolomite samples from the Renison mine area of Tasmania, Australia were analyzed by electron paramagnentic resonance spectroscopy (EPR), Atomic Absorption and Mass Spectrometer to identify alteration related to mineralisation. The least-altered dolomite samples, which are not effected by circulation of diagenetic and hydrothermal fluids are characterised by low Mn and Fe and heavier oxygen isotope values. In contrast, the altered samples are characterised by high Fe and Mn and light oxygen isotope values. Significant differences in EPR intensity exist between the unaltered and altered dolomite samples. The highly altered samples close to the mineralised area are characterised by strong EPR intensities of Mn^{2+} sextets observed at magnetic flux density sweeps over 326.3 ± 2500 mT and 326.3 ± 1000 mT, which correspond to the higher Mn and Fe and lighter oxygen isotope values. In contrast, in the least-altered dolomites, away from the mineralised area, the EPR intensity and Mn and Fe concentrations are low and oxygen isotope values are more enriched.

Introduction

This study attempts to determine whether the combined EPR responses and geochemical characteristics of carbonate rocks in the Renison tin mine are related to alteration and tin mineralisation and to check whether the EPR response of carbonates can be used as an exploration tool in the search for mineral deposits. EPR spectroscopy is a fast technique; the measurement of a spectrum takes only 4 minutes.

Keywords: EPR; Rock geochemistry; Mineral exploration

Several studies investigated the relationship between EPR signals and gold mineralisation in quartz [1-6]. Quartz with anomalous paramagnetic properties or anomalously high amounts of specific impurities is often related to mineralization [7]. Recently, integrated methods of EPR, together with geochemical signatures, have been used to distinguish between gold bearing and barren jasperoids in Carlin, Nevada, USA [8] and in massive sulphide exploration in the Rosebery mine area, western Tasmania, Australia [9]. Xu and Van Moort [6] subsequently studied the character of EPR spectra of regolit samples from a gold

prospect in the Tanami Desert, Northern Territory (Australia) to distinguish between mineralised and non-mineralised zones.

In this preliminary study, it was found that this integrated method is useful in the search for mineral deposits in carbonate rocks. It was also found that highly altered dolomites are more paramagnetic than less-altered ones.

Regional Geology

Renison, the largest primary tin producer in Australia, is situated at Renison Bell, Tasmania, at a latitude of 41° 47′ S and a longitude of 145° 26′E (Fig. 1). The Renison mine sequence consists mainly of the Neoproterozoic Success Creek Group and the Crimson Creek Formation. The Renison deposits at the mine area occupy the upper part of the Success Creek Group and the

lower part of the Crimson Creek Formation (Fig.2). The success Creek Group consists of thick siliciclastic facies hosting the No. 2 and No. 3 dolomite horizons [10]. The Success Creek Group is conformably followed by a thick Succession of relatively shallow marine carbonates, siliciclastics and volcaniclastic of the Crimson Creek Formation [11]. The Crimson Creek Formation hosts the No. 1 dolomite horizon [10].

Carbon isotope chemostratigraphy supports the Neoproterozoic age of about 570-820 Ma for the Crimson Creek Formation and the Success Creek Group respectively [12].

The Renison tin mine sequence is interrupted by the Devonian Pine Hill Granite [13, 11], (Fig.2). Regional fault structures at Renison are attributed to a tensional regime associated with the emplacement of the Pine Hill
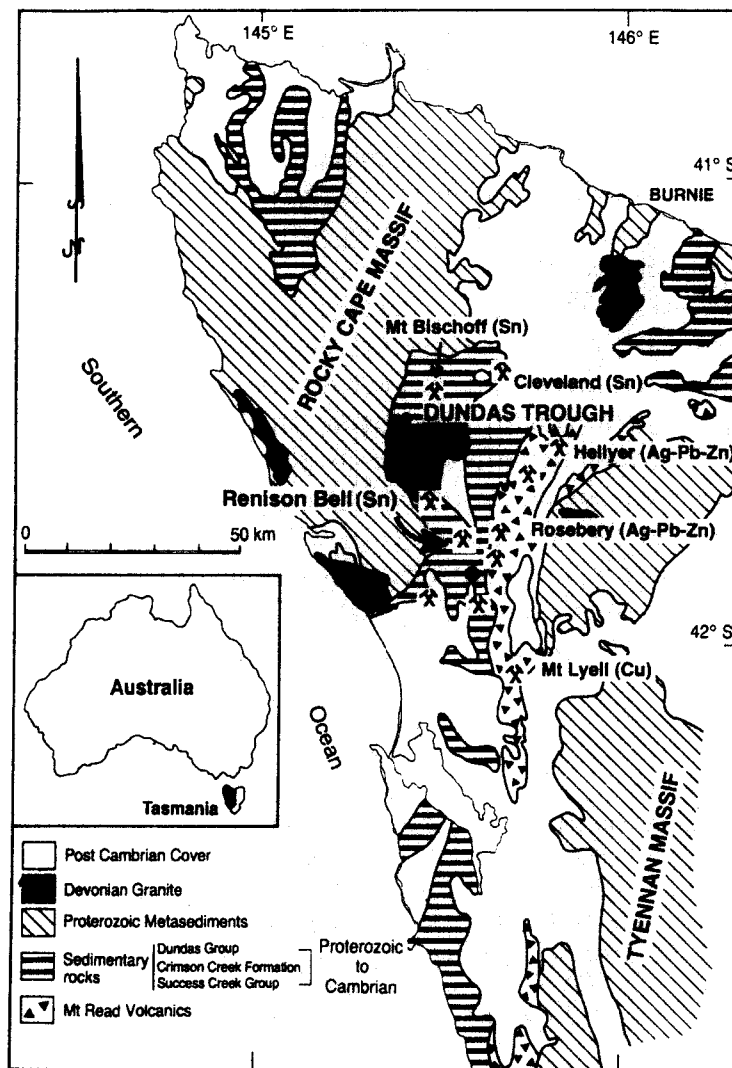

Figure 1. Regional geology of western Tasmania (Australia) and the location of the Renison tin mine
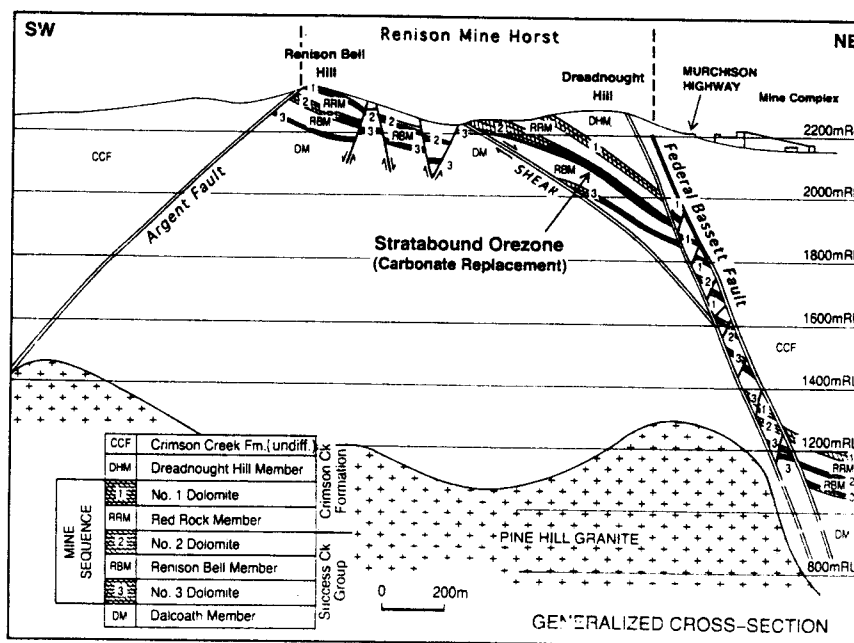
Figure 2. Schematic cross section of the Renison deposit showing the distribution of stratabound carbonate-replacement tin deposit (modified after Cannard) [13]

Granite (Fig. 2), [14]. The Mine Sequence is disrupted by at least three major groups of mineralised faults formed during the forceful emplacement of the Pine Hill Granite (Fig. 2), [11].

Mineralisation resulted from the passage of hydrothermal fluids, sourced from the Devonian Pine Hill Granite, through major fault structures, partially replacing the three dolomite horizons and forming a cassiterite-rich pyrrhotite orebody [11,15]. The Federal-Bassett Fault (FBF) was the primary structural control on the distribution of ascending magmatic hydrothermal fluids at Renison [11,15].

Petrographic Study

Dolomite fabrics identified by a normal petrographic microscope have been classified according to crystal size and mode of origin. These include: 1) very finely crystalline dolomite (dolomicrite, ~ 5 μm to ~ 16 μm); 2) finely crystalline dolomite (dolomicrosparite, ~ 16μm to ~ 62 μm); 3) medium crystalline dolomite (dolosparite, ~ 16 μm to ~ 250 μm); 4) coarse to very coarsely crystalline dolomite (vein dolomite, > 250 μm).

Dolomicrites are the most abundant dolomite types away from the mineralised area. Dolomicrites are considered marine cements as the original depositional and early diagenetic textures are well preserved and there is no evidence of a replacement origin. Dolomicrosparites are formed through dolomicrite recrystallisation [15, 16].

In a few samples, the clots of original dolomicrite within dolomicrosparite support diagenetic recrystallisation. Dolosparites which fill pore spaces, cavities and vugs are referred to as late diagenetic dolomites. Vein dolomite is in sharp contact with the host wall rock and generally cuts original, early and late diagenetic dolomites. The vein dolomites are volumetrically very important in mineralised areas compared to the unmineralised areas. Detailed petrographic studies of these dolomite types have been discussed by Adabi [16].

Principles of EPR Spectroscopy

Paramagnetism is caused by the alignment of atomic dipoles in a magentic field and occurs in all atoms, ions and molecules with odd numbers of electrons, colour centres (when unpaired electrons are trapped in the crystal lattice) and metallic compounds [17]. Paramagnetism is much weaker than ferromagnetism and appears if a material is placed in a strong external magnetic field [9]. Electron paramagnetic resonance (EPR) is also known as electron spin resonance (ESR). This technique, which was discovered by Zavoiski in 1945, is based on the resonant absorption of microwaves by paramagnetic substances applied by an external magnetic field.

EPR is based on the property of electrons and the resonance of unpaired electrons in a sample. It is measured in an EPR spectrometer at specific levels of a strong, applied magnetic field while a microwave of constant

frequency and intensity is simultaneously applied [9].

The EPR signature of quartz has been extensively studied by many authors (see reviewed paper by Weil [18], due to its technological and geological importance. Quartz is an ideal mineral for EPR study because its unpaired electrons are associated with the presence of electron donors like Al, Fe, Ga and electron acceptors such as Ge and Ti, replacing Si [9, 18]. The Perfect quartz does not have unpaired electrons and thus gives no EPR signal [18]. Near ore bodies, however, the quartz contains numerous atoms out of place and various substitutional and interstitial impurities, some of which are inherently paramagnetic (e. g., Fe^{3+}), [19]. Consequently this causes a considerable amount of lattice defects and gives paramagnetism [6, 18, 19]. In carbonate rocks, particularly dolomites, there are also several substitutional elements such as Mn and Fe which are paramagnetic.

Detailed descriptions of the EPR theory and application in geology are given by Pilbrow [20] and Calas [17].

Instrumentation

An EPR spectrometer consists of four major subsystems. These are: 1) a microwave bridge and sample resonator (sample cavity); 2) two large electromagnets and power supply with field control and field sweep; 3) detector and signal pre amplifier followed by phase-sensitive detection

(PSD) at the magnetic field modulation frequency, a cathods ray oscilloscope (CRO), recorder display, and 4) computer acquisition and data processing (Fig. 3).

A microwave source, usually a klystron, is connected to a reference arm type bridge. A microwave unit generates stable microwave radiation by an external constant frequency generator of klystron or Gunn diode. The klystron, which is a source of microwave radiation, is operated within a high frequency range. The microwave radiation passes through the attenuator and circulator and into the sample cavity (resonator) where the powder sample is located. The sample cavity is located in the centre of a strong homogeneous magnetic field between two large electromagnets. It includes the components to hold the sample and to direct and control the microwave beam to and from the sample.

The intensity of the outcoming microwave from the resonance cavity is measured by means of a circulator and a detector. The EPR intensities are measured as the first derivative of the absorption of microwave energy coming back from the sample against the intensity of the magnetic field. There were two measurements: 1) measurement at magnetic flux density sweeps 326.3 ± 2500 mT, and (2) measurement at magnetic flux density sweeps 326.3 ±1000 mT. The intensity of the EPR signal (measured as peak height of absorption peak) is proportional to the total number of unpaired spins in the centres.
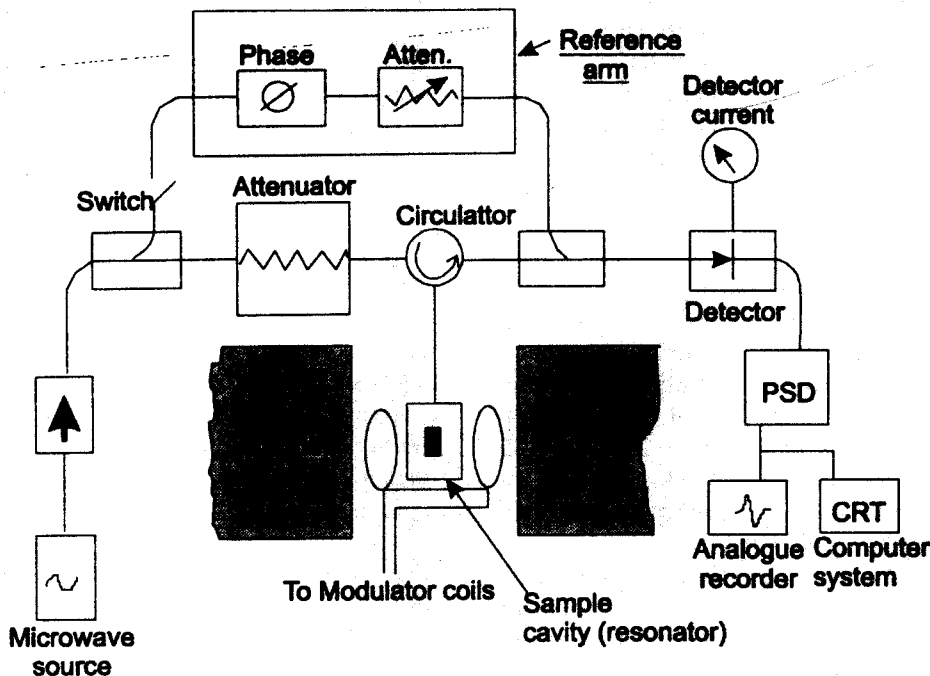

Figure 3. Block diagram of a representative modern homodyne EPR spectrometer. The reference arm controls the phase and amplitude of the signal (modified after Pilbrow) [20]

Manganese Centre

Well developed Mn^{2+} sextets are common in most dolomite units of the Renison mine area (Fig.5). The centre is very important for the present study as Mn^{2+} sextets are significant characteristics of the altered rocks, particularly close to the mineralisation. These Mn signals are related to the Mn content of the dolomite samples. The more altered dolomite samples in the Renison mine area have higher Mn and Fe contents compared to the least altered ones.

Sample Preparation and Instrument Setting

In this study, twenty six surface drill core samples (S705, S835, S594) and twenty underground samples were used (Fig.4). Dolomite was sampled for chemical analysis using a dental drill under a binocular microscope. Fifteen mg of dolomite powder were allowed to react with anhydrous phosphoric acid (H_3PO_4) in reaction tubes under a vacuum for 24 hours. The evolved Co_2 from each sample was analysed for oxygen by using a Micromass 602 D Mass Specrtometer at the Central Science Laboratory, University of Tasmania (Australia). Oxygen isotope values of dolomite samples were corrected by subtracting 1‰ from each measured oxygen value to standardise values. These values are expressed in conventional per mil notation relative to PDB standard. The precision of data established with duplicate analyses for oxygen is ± 0.1‰. Portions of the powders used for isotope analyses were dissolved in 1M HCl for two hours and analysed for Mn and Fe by

atomic absorption spectrophotometry. The detection limits are ± 5 ppm for Mn and Fe.

A measure of the paramagnetism of all dolomite powder samples was obtained by using an analogue JEOL JES FE3X EPR spectrometer at the Central Science Laboratory of the University of Tasmania, Australia. The 100 mg dolomite powder samples were placed in a Wilmad annealed silica EPR tube, inserted in a sample cavity, and subjected to a microwave power frequency of 9.15 GHz (X-band), 10mW microwave power modualtion frequency at 100 KHz, magnetic field center with a median value of 3265.3 mT amplitude at 1×10 and sweeping (scan) ranges of 326.3 ±1000 mT and 326.3 ±2500 mT. The first spectrum (326.3±1000 mT) provide details of the shape of the peak. All the rock powder samples were run at room temperature. The EPR intensities were measured as the peak height in centimeters between the two turning points of the first derivative signal present in the spectrum (Fig. 5). This measurment provides an approximation of the intensity of the absorption of the microwave power at resonance [8].

Relation between EPR Intensities and Geochemical Response

There are two types of investigation in this study. The first one is the correlation between EPR intensities and element concentrations (Mn and Fe) and the other, the relationship between EPR intensitites and oxygen isotope values in different dolomite types.
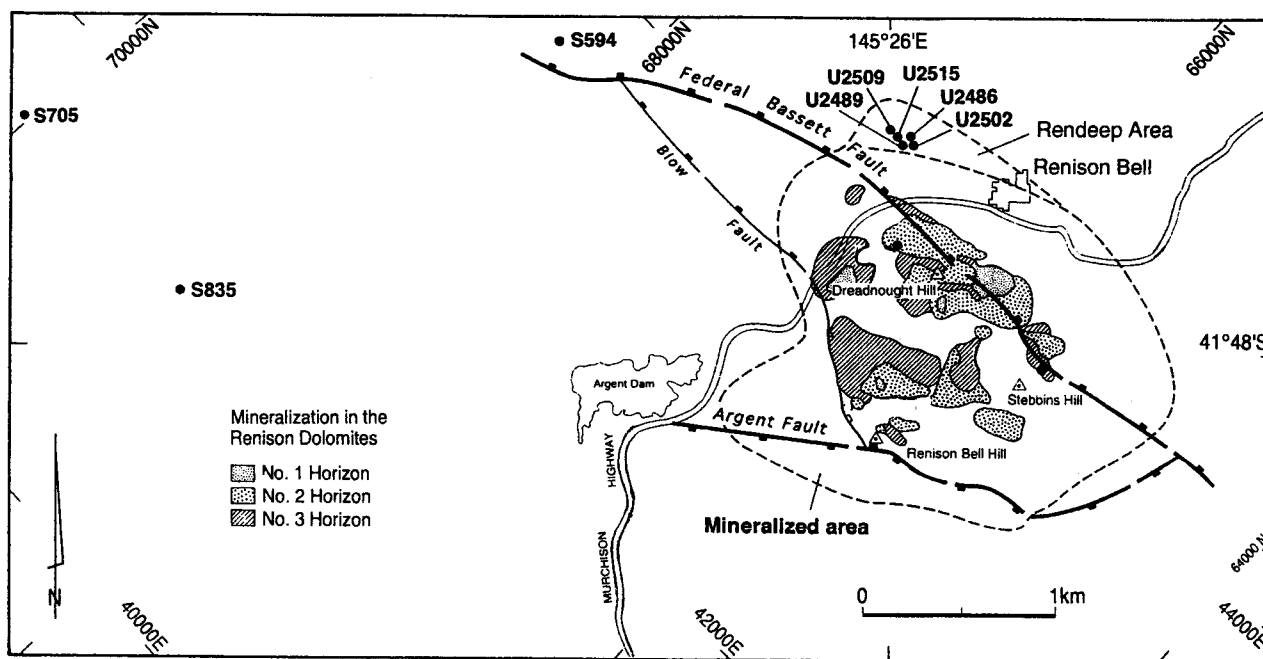

Figure 4. Diamond drill hole locations and underground samples sites at Renison
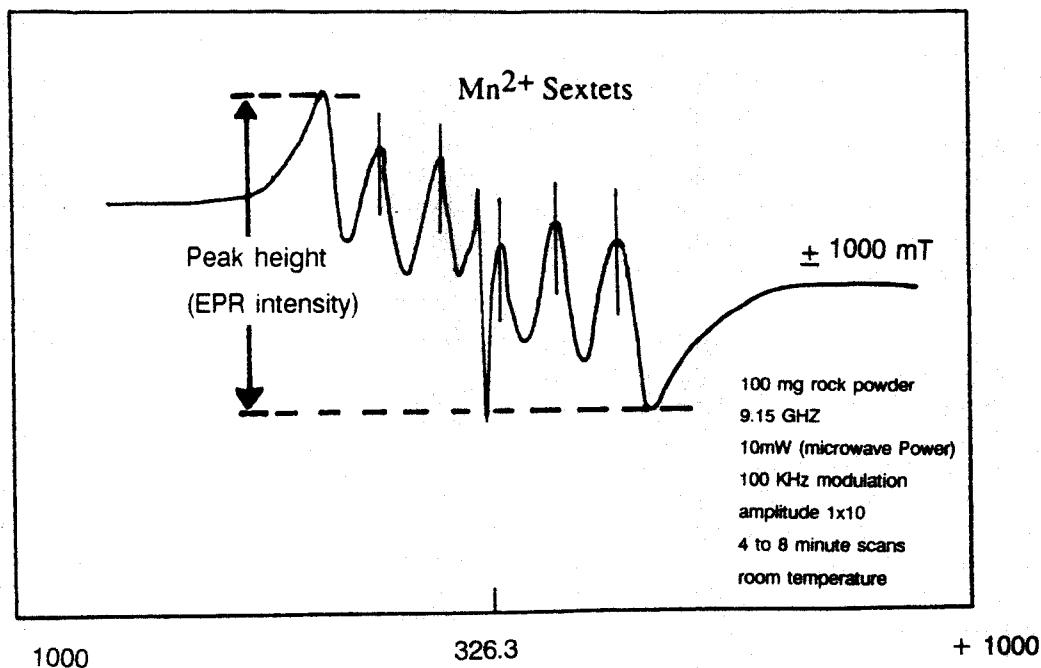
Figure 5. The measurement method of EPR intensity

Covariation of Mn and Fe with EPR

Mn and Fe concentrations in carbonates vary with carbonate mineralogy, fluid composition, partition coefficients and oxidising and reducing conditions [21,22, 23, 24].

Mn and Fe concentrations are low in seawater whereas they are high in non-marine fluids [21]. Since the distribution coefficients for both Mn and Fe in dolomite are greater than unity [21, 23], high Mn and Fe concentrations can occur in dolomite. The incorporation of Mn and Fe into dolomite is related to prevailing aerobic-anaerobic conditions druing alteration. Aerobic conditions prevent the substitution of Mn and Fe for Mg in dolomite, whereas anaerobic conditions favour large concentrations of these elements [21,25]. Therefore, high concentrations of both Mn and Fe in dolomite may be due to carbonate alteration under anaerobic conditions.

In dolomicrite, Mn ranges from 120-385 ppm; average 231 ppm and Fe ranges from 1500-6673 ppm; average 3207 ppm (Table 1). The relatively high iron enriched dolomicrites, compared to the modem ones are due to the availability of iron in the basin during dolomicrites formation as evidenced by the relatively thick sequence of a banded iron formation above and below the dolomite horizons in the Renison sequence [16].

The Fe and Mn concentrations in recrystallised dolomicrosparites are higher than dolomicrites (Mn ranges from 329-3958; average 1393 ppm and Fe ranges from 2980-23337; average 14017 ppm).

The Fe and Mn concentrations in dolosparites are higher than dolomicrite and dolomicrosparite (Mn ranges from 751-45455; average 2790 ppm and Fe ranges from 13082 -25219; average 18657 ppm).

Vein dolomites contain the highest Mn (ranges from 2157-6158; average 4046 ppm) and Fe (ranges from 24945-27573; average 26192 ppm) concentrations (Table 1).

The increase of Fe and Mn concentrations from least-altered dolomite to altered dolomite is due to the composition of fluids altering the dolomite. As the Fe and Mn concentrations are low in marine fluids [22,23], the increase of Fe and Mn values from least-altered (dolomicrite) to altered dolomite is due to reaction with non-marine fluids. Petrographic features, major and minor elements, together with oxygen and carbon studies, indicate that the Renison dolomites have been altered mainly by magmatic hydrothermal fluids [15,16]. Since the incorporation of Fe and Mn in carbonate increases from oxidizing to reducing conditions [21,26], the high concentrations of Fe and Mn in the dolomite studied may also be due to reducing conditions during alteration and formation of diagenetic dolomite [25].

The EPR intensity measured in the least-altered dolomicrite has a range of 1.1 to 6.5 cm with an average value of 3.57 cm (Table 1). At a first approximation, peak height correlates positively to the integrated value of the first irrevative peak.

The EPR intensity is stronger in more altered rocks such as dolomicrosparite, dolosparite and vein dolomite.

Table 1. Dolomite type, EPR intensity, minor elements and oxygen and carbon isotope values away from and close to the Rension mine area

Sample No	Dolomite type	EPR intensity (cm)	Mn ppm	Fe ppm	$\partial 180$ PDB
S 705-166m	dolomicrite	6.5	385	2812	-1.8
S 705-166.2	dolomicrite	2.7	234	2442	-0.7
S 705-169.8	dolomicrite	2.9	209	1600	-1
S 705-171.7	dolomicrite	6	306	3430	-1.5
S 705-172	dolomicrite	4.2	214	1548	-1.4
S 705-172.8	dolomicrite	3.9	194	3757	-0.3
S 705-172.8 C	dolomicrite	3.7	240	2825	-0.8
S 705-179.1	dolomicrite	3.1	268	6673	-1.3
S 705-179.8	dolomicrite	3.7	304	2204	-1.6
S 705-180.4 B	dolomicrite	3	120	2882	-1.4
S 705-181.4	dolomicrite	6.1	238	5756	-2.1
S 705-183	dolomicrosparite	5.4	333	2980	-2.8
S 705-184 B	dolomicrite	4.12	252	4118	-1.9
S 705-187	dolomicrosparite	2.6	333	10015	-2.9
S 705-187.4 A	dolomicrite	1.6	157	4782	-0.8
S 705-187.5 B	dolomicrite	1.1	160		-1.8
S 705-189.8	dolomicrosparite	4.7	329	4658	-4.3
S 705-202.3	dolosparite	5.4	751	20233	-7.8
S 705-245.6	dolomicrosparite	3.5	343	13473	-3.1
S835-201.6	dolomicrite	3	282	2471	-0.7
S835-204.5	dolomicrite	1.5	132	4008	0.1
S835-213.4	dolosparite	13.3	3235	18940	-8.9
S835-272.3	dolomicrosparite	6.3	1378	14620	-5.2
S594-233.5 B	dolosparite	12.7	1690	13082	-7.2
S594-236.8	dolosparite	12.6	3836	19421	-8.2
S594-279	dolomicrosparite	8.7	1765	17178	-6.9
U2486-269	dolosparite	21.4			-15.1
U2489-298.1	vein dolomite	16			-12.5
U2489-298.1	dolomicrosparite	13.2	2253	19323	-7.2
U2489-311	dolosparite	18.7	2728	24701	-8.6
U2502-269.95	dolosparite	12.3	4503	25219	-8.4
U2502-270	dolomicrosparite	17.2	3958	23337	-7.3
U2502-270.15	dolosparite	15	4555	22294	-8.1
U2502-270.15	vein dolomite	16.48	3823	27573	-8.6
U2509-312.5	dolomicrosparite	12	1560	15242	-6.5
U2509-312.6	dolomicrosparite	11.6	1760	15471	-6
U2515-314.4	vein dolomite	30.2	6158	26058	-9
U2515-314.4	dolosparite	25.6	4303	22177	
U2515-319.7	dolomicrosparite	13	1584	15617	-6.9
U2515-319.7	dolomicrosparite	14.2			-3.8
U2515-323.2	dolomicrosparite	5.8	1970	14278	-5.5
U2515-323.2	vein dolomite	15.3			-6.7
U2515-335.4 C	dolosparite	15.2	2297	20500	-7.4
U2515-335.4	vein dolomite	23	2157	24945	-9
U2515-341.4 A	dolomicrosparite	16.1	1751	23000	-5.7
U2515-341.4 B	dolomicrosparite	16.6	1574	21058	-5.5

In recrystallised dolomicrosparite, the EPR intensity ranges from 2.6 to 17.2 cm with an average value of 10.1 cm (Table 1). The EPR intensity measured in dolosparite is stronger than dolomicrite and dolomicrosparite and ranges from 5.4 to 25.6 cm with an average value of 15.2 cm. In vein dolomite, the measured EPR intensity shows the highest value compared to other dolomite types and ranges from 15.3 to 30.2 cm with an average value of 20.2 cm (Table 1).

The EPR intensity measured in all dolomite samples correlates positively with Fe and Mn (Fig. 6). The highly altered samples which contain higher Fe and Mn values exhibit strong EPR intensity. Such relationships indicate that the EPR response in the rocks is related to wall-rock alteration. However, the least-altered host rock (dolomicrite) exhibits different behaviour and correlates negatively with Fe and Mn. The correlation coefficient (r^2 values) in all samples are significant (~0.7).

The EPR intensities measured at magnetic flux density sweeps over 326.3 ± 1000 mT and ± 2500 mT in dolomicrite, dolomicrosparite, dolosparite and vein dolomite are shown in Figures 7A-D, 7a -d.

Covariation of Oxygen Isotope and EPR

The finely crystalline dolomicrite have a narrow range of oxygen isotope values, from +0.3 to -2.9‰ PDB, with an average of -1.1‰ PDB (Table 1). The oxygen isotope values in dolomicrites are significantly heavier than those of other dolomite types (Fig. 6). Dolomicrite is interpreted as the earliest formed dolomite in the Renison mine area [15,16].

The EPR intensity in the least-altered dolomicrite is very low with an average value of 3.57 cm. The very low EPR intensity and heavy oxygen isotope values are possibly indicative of early diagenetic dolomitisation.

The oxygen isotope values in dolomicrosparite range from -2.8 to -7.3‰ PDB with an average of -5.5‰ PDB. These oxygen isotope values are lighter than those in dolomicrite due to recrystallisation.

The EPR intensity in recrystallised dolomicrosparite is higher than that in dolomicrite but lower than other dolomite types (Table 1).

The oxygen isotope values in dolosparite show a wide range, from -7.4 to -15.1‰ PDB with an average of -8.8‰ PDB. The oxygen isotope values in dolosparite are lighter than dolomicrite and dolomicrosparite (Fig. 6) due to late diagenetic dolomitisation.

The EPR intensity in dolosparite shows a wide range with an average value of 15.2 cm. The very high EPR intensity which corresponds to the lightest oxygen isotope value (Fig. 6) may indicate post depositional diagenetic modification, possibly by non-marine fluids. The common diagenetic alteration processes leading to oxygen isotope
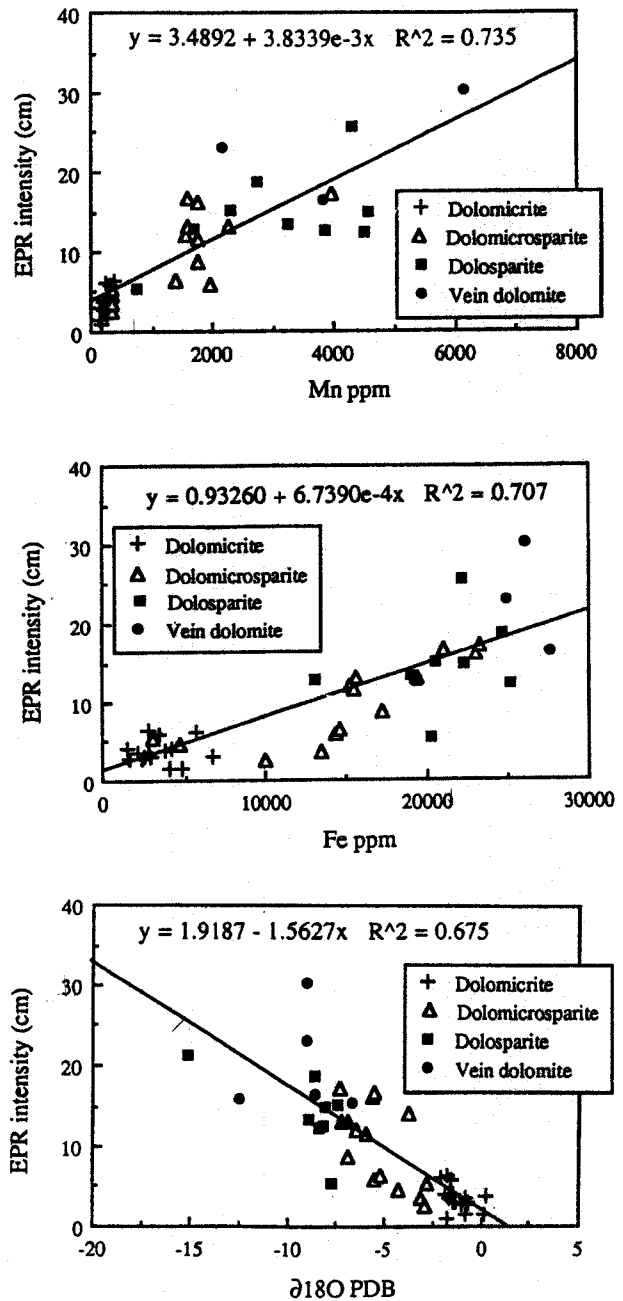

Figure 6. Variation of Mn, Fe and oxygen isotope values versus EPR intensity

depletion are either meteoric water and/or fluids at elevated temperatures. Detailed diagenetic fluid studies in the Renison mine area indicate that hydrothermal fluids were responsible for carbonate alteration [15,16]

The oxygen isotope values in vein dolomite range from -6.7 to -12.5 ‰ PDB with an average of -9.2‰ PDB. The vein dolomite has the lowest average oxygen
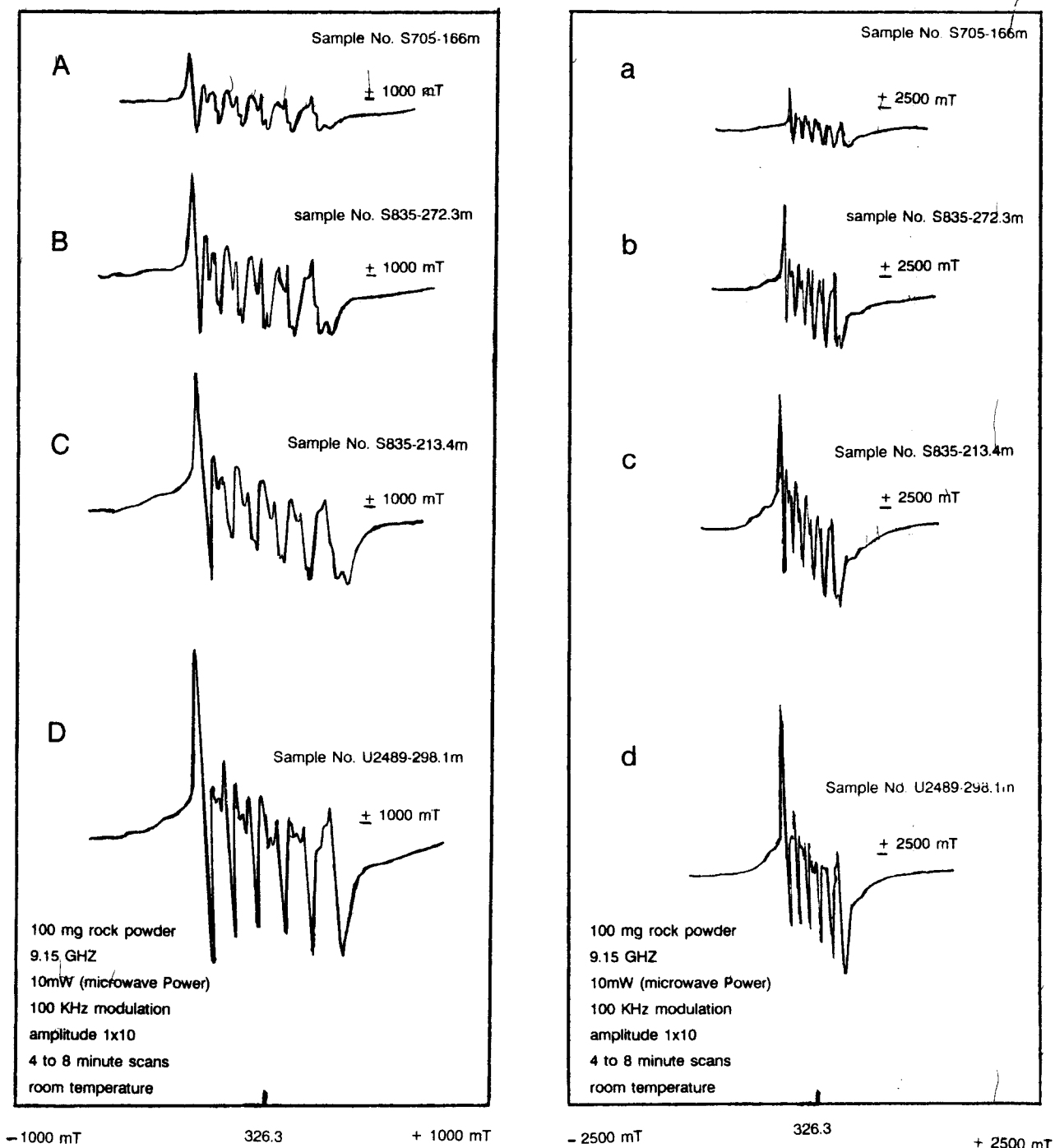
Figure 7. Comparison of EPR signal in least-altered dolomicrite (A), moderately altered dolomicrosparite (B), highly altered dolosparite (C), and vein dolomite (D). Note that the EPR spectrum is stronger in more altered dolomite rocks. EPR spectrum measured at magnetic flux density sweeps 326.3 ±1000 mT showing more detail of the peak shape compared to 326.3 ±2500 mT

isotope value compared to other dolomite types.

The EPR intensity measured in vein dolomite shows the highest value compared to other dolomite types.

Distinction between Mineralised and Unmineralised Areas

The EPR intensities, Mn and Fe concentrations and oxygen isotope values are very much related to the tin mineralisation in the samples studied. The results show that underground samples (U2486-U2515) which are very close to the orebody (distance range from 1 to 110m) are more altered and hence, more affected by hydrothermal alteration. In contrast, the surface samples (S705, S835, S594) which are located within a few km of the mine area (Fig. 4) are less altered and hence least-affected by hydrothermal alteration.

The EPR intensity and the Mn and Fe content increase while the oxygen isotope values decrease toward the ore body. The EPR intensity values are very low (average 5.1 cm) in surface samples while the highest EPR peak height (average 16.4 cm), occurs in the underground samples close to the mineralised area. Low average Mn (680 ppm) and Fe (7436 ppm) concentrations can be found in the surface samples away from mineralised areas while the very high Mn (average 2933 ppm) and Fe (21300 ppm) contents are present in the underground samples close to the orebody. Oxygen isotope values are more enriched (average, -2.9‰ PDB) in surface samples while the oxygen isotope value decreases (average, -7.8‰ PDB) in underground samples close to the orebody.

Mineralogical alteration such as silicification, sericitisation and chloritisation are predominant in the highly altered dolomite samples close to mineralisation [16].

Therefore, the EPR signal, Mn and Fe concentrations and oxygen isotope values can be used to discriminate the mineralised from the unmineralised area A noticeable feature in samples close to the orebody is that the EPR intensity is more than 10 cm (except one sample). This corresponds well with the highest Mn and Fe concentrations and lightest oxygen isotope values. The EPR intensity greater than 10 cm close to the ore body in the Rosebery mine, western Tasmania, Australia was recently reported by Aung Pwa [9]. The results of EPR work by Xu and van Moort in the goldfields of western Australia show that the highest EPR value, ranging from 5-10 cm, occurs over the mineralised zone (personal communication, 1997). In quartz and dolomite, unpaired electrons are accummulated during ore formation. If samples were not ore related, there will be no such unpaired electrons accumulating and no alignment of electrons will happen under a magnetic field, i.e. only ore related minerals or components are paramagnetic

substances, which can be converted to paramagnetic form [6].

Conclusion

The EPR response of dolomite rock powder samples exhibits a significant relation to tin mineralisation and rock alteration. Although this is a preliminary study to examine whether the EPR response of carbonate rocks is useful in the search for ore deposit, the results are encouraging for further study.

There are significant differences in the intensities of the EPR signal between altered and least-altered dolomite samples. The more altered rocks, which are near or around mineralisation, are characterised by strong EPR signals (normally more than 10 cm), whereas the least-altered rocks (away from the mineralisation) exhibit weak signals. The majority of the spectra are associated with well developed Mn^{2+} sextets, but some do not show significant Mn^{2+} sextets. The Mn signals are related to the Mn content of the dolomite samples.

The EPR intensities remarkably display a strong positive correlation with elements such as Mn and Fe and negative correlation with oxygen isotope values. These are con sidered to be related with wall rock hydrothermal alteration and tin mineralisation.

The dolomite samples with more than 10 cm EPR intensity, high Mn and Fe concentrations and very light oxygen isotope values are considered to be important for exploration study.

The results of the EPR study in the Renison mine area support an application of the combined EPR and geochemical methods to mineral exploration and distinction between least-altered and more altered rocks, which are related to hydrothermal alteration and tin mineralisation.

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