ZONED MICHENERITE–TESTIBIOPALLADITE FROM KAMBALDA, WESTERN AUSTRALIA

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ABSTRACT

Euhedral grains of michenerite–testibiopalladite occur as inclusions in altaite from a telluride-bearing quartz-carbonate vein that cuts nickel-iron sulfide ore at Lunnon Shoot, Kambalda. Grains vary in size from 50 to 200μm and consist of concentric compositional zones that show a progressive but step-like decrease in antimony and increase in bismuth from a core of testibiopalladite to a margin of michenerite. This is the first reported occurrence of testibiopalladite outside China. Electron microprobe analyses of three compositional zones show the variation from $(Pd_{0.97}Pt_{0.01}Ni_{0.01})_{\Sigma 0.99}(Bi_{0.34}Sb_{0.63}Ag_{0.01})_{\Sigma 0.98}Te_{1.04}$, to $(Pd_{0.97}Pt_{0.02}Ni_{0.01})_{\Sigma 1.00}(Bi_{0.56}Sb_{0.38}Ag_{0.01})_{\Sigma 0.95}Te_{1.06}$, and to $(Pd_{0.96}Pt_{0.02}Ni_{0.01})_{\Sigma 0.99}(Bi_{0.74}Sb_{0.18}Ag_{0.01})_{\Sigma 0.93}Te_{1.08}$. Line scans demonstrate an almost complete range of compositions between previous data for michenerite and testibiopalladite. Hardness determinations give VHN 371 for testibiopalladite and 333 for michenerite. Other minerals in the vein are hessite, volynskite, rucklidgeite, melonite, galena, chalcopyrite and gold.

SOMMAIRE

Des cristaux idiomorphes de michenerite–testibiopalladite se trouvent en inclusions dans l'altaïte dans un filon de quartz et carbonates contenant des tellurures qui traverse la zone minéralisée en sulfures de nickel et de fer, à Lunnon Shoot, Kambalda. Ces cristaux, dont la taille va de 50 à 200 μm, sont formés de zones concentriques qui, du centre à la périphérie, montrent un appauvrissement en antimoine et un enrichissement en bismuth, passant ainsi de la testibiopalladite à la michenerite en composition. C'est la première découverte de testibiopalladite hors la Chine. Des analyses à la microsonde de trois zones indiquent une transition de $(Pd_{0.97}Pt_{0.01}Ni_{0.01})_{\Sigma 0.99}(Bi_{0.34}Sb_{0.63}Ag_{0.01})_{\Sigma 0.98}Te_{1.04}$ à $(Pd_{0.97}Pt_{0.02}Ni_{0.01})_{\Sigma 1.00}(Bi_{0.56}Sb_{0.38}Ag_{0.01})_{\Sigma 0.95}Te_{1.06}$ et à $(Pd_{0.96}Pt_{0.02}Ni_{0.01})_{\Sigma 0.99}(Bi_{0.74}Sb_{0.18}Ag_{0.01})_{\Sigma 0.93}Te_{1.08}$. Les parcours du balayage indiquent une gamme presque complète de compositions entre les données antérieures pour la michenerite et la testibiopalladite. La dureté de la testibiopalladite est de 371 VHN, celle de la michenerite, de 333. D'autres minéraux se trouvent dans le filon; en voici la liste: hessite, volynskite, rucklidgeite, mélonite, galène, chalcopyrite et or.

(Traduit par la Rédaction)

INTRODUCTION

Michenerite (PdBiTe) is a rare palladium mineral that occurs in association with Ni–Fe–Cu sulfide mineralization at Sudbury (Cabri et al. 1973, Cabri & Laflamme 1976), Monchegorsk and Oktyabrskoe, USSR (Genkin et al. 1963, 1972), Hitura, Finland (Häkli et al. 1976), Merensky Reef, South Africa (Vermaak & Hendriks 1976) and from localities in China (Huang et al. 1974). It is also present in the Witwatersrand gold ores (Feather 1976) and occurs with hydrothermal copper ores in Wyoming (McCallum et al. 1976).

The mineral was first described by Michener (1940) as Pd_2Bi_3, a 6.65Å, but was later named and redefined as $PdBi_2$ by Hawley & Berry (1958). Studies by Genkin et al. (1963) showed that michenerite grains from Monchegorsk have

compositions $Pd_{0.44}Pt_{0.18}Bi_{0.81}Te_{1.18}$ and $Pd_{0.72}Pt_{0.21}Bi_{0.98}Te_{1.02}$, with a "best" formula $Pd_{0.75}Pt_{0.25}BiTe$. Genkin *et al.* (1972) also described michenerite from the Oktyabrskoe deposit having a formula $Pd_{1.15}(Bi_{0.86}Te_{1.14})_{\Sigma2.00}$. Cabri *et al.* (1973) analyzed a number of specimens from the Sudbury district and, as a result, redefined michenerite as PdBiTe.

Substitutions in michenerite can cause departures in composition from the defined formula; these are summarized as (Pd,Pt,Ni)(Bi,Sb,Te)(Te,Bi), or can be expressed as $(Pd,Pt,Ni)(Bi,Sb,Te)_2$, as suggested by Genkin *et al.* (1972). Substitutions of Ag for Pd and Pb for Bi have also been reported but are of less certain status. The analyses of Sudbury material on which Cabri *et al.* (1973) based their redefinition of the mineral are all close to the ideal formula, but do show some substitution of Sb for Bi to give a range of calculated formulae from PdBiTe to $Pd(Bi_{0.89}Sb_{0.12})Te_{1.01}$.

In a subsequent study, Cabri & Laflamme (1976) delineated two compositional groups of michenerite on the basis of Pt content. The low–Pt group showed substitution of Ni for Pd and Sb for Bi up to $(Pd_{0.95}Ni_{0.05})(Bi_{0.80}Sb_{0.19})Te$. Grains of antimonian michenerite showed a substitution of Te for Bi to give calculated formulae with tellurium contents of up to $Te_{1.16}$. This substitution of Te for Bi, and to a lesser extent Bi for Te, had been predicted in experimental studies by Cabri *et al.* (1973) and Hoffman & MacLean (1976). Hoffman & MacLean consider the stability field of michenerite above 450°C to consist of a narrow solid solution in which Bi and Te may substitute to give compositions between $Pd_{0.99}Bi_{0.78}Te_{1.22}$ and $Pd_{0.95}Bi_{1.11}Te_{0.94}$. A michenerite from China (Huang *et al.* 1974) has the formula $PdBi_{0.76}Te_{1.24}$ and thus exceeds the limit of Te–for–Bi substitution found experimentally.

The platinian michenerites from Sudbury vary in composition between 2.1% to 13.9% Pt by weight, with an extreme composition of $Pd_{0.66}Pt_{0.34}Bi_{1.05}Te_{0.96}$. They are thus even more platinum-rich than those described by Genkin *et al.* (1963) from Monchegorsk (8.4 to 9.3 wt.% Pt). Hardness values for platinian michenerites do not differ from normal michenerites, though reflectance values are lower at the shorter wavelengths (Cabri & Laflamme 1976).

A silver content of 1.8 wt. % was reported in michenerite from Oktyabrskoe; silver was described as replacing palladium (Genkin *et al.* 1972). Cabri *et al.* (1973), however, point out the possibility of interference between $PdL\beta$ and $AgL\alpha$, not discussed by Genkin *et al.* Minor lead (1.9 wt. %) reported in michenerite from

Oktyabrskoe (Kovalenker 1977) is assumed to substitute for bismuth. The analysis of michenerite from Hitura, Finland (Häkli *et al.* 1976) shows it to be close to ideal, with minor substitution of Ni for Pd and Sb for Bi. An antimony-rich michenerite, $Pd_{1.05}(Bi_{0.55}Sb_{0.39})Te_{1.01}$, was reported from the Witwatersrand (Feather 1976). It is of particular relevance to this study, as its composition is intermediate between michenerite and testibiopalladite.

Testibiopalladite (PdSbTe) is the antimony analogue of michenerite. The mineral was first described from two Cu–Ni sulfide deposits in China (P.M.M.R.G. 1974; summary in Fleischer *et al.* 1976). In southwestern China a mineralized serpentinite, emplaced into metamorphosed Permian rocks, contains testibiopalladite inclusions in an assemblage comprising gersdorffite, cobaltite, pyrrhotite, chalcopyrite and pentlandite. The other locality is in northeastern China where the mineral occurs as a relatively common constituent of a sulfide-bearing clinopyroxenite that intrudes Permian sandy shales and volcanic rocks. Electron microprobe analyses indicate the range of compositions $(Pd_{0.89-0.99}Ni_{0-0.06})(Sb_{0.63-0.97}Bi_{0-0.36}Te_{0.92-1.06})$.

Physical properties of previously described michenerite and testibiopalladite are compared in Table 1.

DESCRIPTION OF THE KAMBALDA OCCURRENCE

The Kambalda nickel deposits were discovered in 1966, and since then have been Australia's principal nickel producers; they were described most recently by Ross & Hopkins (1975). Platinum-group elements occur in the ores (Keays

TABLE 1. PHYSICAL PROPERTIES OF DESCRIBED[†] MICHENERITE AND TESTIBIOPALLADITE

	MICHENERITE	TESTIBIOPALLADITE
Composition	PdBiTe	PdSbTe
Space Group	$P2_13$	$Pa3$
Cell parameter	a 6.63-6.65	a 6.56-6.58Å
z	4	4
Density	10.0g/cm^3(calc.)	
Cleavage	-	two imperfect
Color	"galena" grey	bright steel grey (yellow-brown tarnish)
Lustre	metallic	metallic
Reflected light	creamish white with grey tinge	white with pale blue or creamy tinge
Anisotropy	isotropic (rarely anisotropic)	isotropic
Reflectances	58.2 (470nm)	54.4 (480nm)
	55.2 (546nm)	54.9 (540nm)
	54.3 (589nm)	54.9 (580nm)
	54.7 (650nm)	54.0 (640nm)
VHN_5		165
VHN_{10}	189*	267
VHN_{25}	311-321	-

†Data on michenerite from Cabri *et al.*(1973), Childs & Hall(1973), and Cabri & Laflamme (1976); data on testibiopalladite from P.M.M.R.G. (1974).
*Michenerite with 8 wt.% Pt.

TABLE 2. ELECTRON PROBE ANALYSES OF MICHENERITE-TESTIBIOPALLADITE

	TESTIBIOPALLADITE CORE 1				CENTRE 2				MICHENERITE RIM 3			
	Wt.%	At.%	Σat=3		Wt.%	At.%	Σat=3		Wt.%	At.%	Σat=3	
Pd	26.6	32.26	0.968		25.3	32.46	0.974		24.1	31.98	0.959	
Pt	0.5	0.33	0.010	0.99	1.0	0.70	0.021	1.00	1.1	0.80	0.024	0.99
Ni	0.1	0.22	0.007		0.1	0.23	0.007		0.1	0.24	0.007	
Bi	18.1	11.17	0.335		28.5	18.61	0.558		36.6	24.73	0.742	
Sb	19.8	20.99	0.629	0.97	11.2	12.56	0.377	0.94	5.3	6.14	0.184	0.93
Ag	0.2	0.24	0.007		0.2	0.25	0.008		0.2	0.26	0.008	
Te	34.4	34.79	1.044	1.04	32.9	35.19	1.055	1.06	32.4	35.85	1.076	1.08
	99.7*				99.2*				99.8*			

* Totals

& Davison 1976) but no systematic study has been carried out to determine their mode of occurrence, apart from microprobe studies associated with routine, generally unpublished documentation of the sulfide mineralogy of the first two nickel orebodies discovered, Lunnon and Durkin Shoots. This work established the presence of sperrylite, palladian melonite and Pd–Bi tellurides of uncertain composition.

Gold and telluride-bearing quartz and quartz–carbonate hydrothermal veins, commonly associated with porphyritic sodic rhyolite and dacite intrusive bodies, are a minor feature of the Kambalda area. Where these veins cut the nickel sulfide mineralization, unusual mineral suites containing lead, bismuth, silver and palladium tellurides have been recorded. In the occurrence described here from Lunnon Shoot an 'opaque' modal analysis determined by point count gave 84.5% altaite, 6.1% rucklidgeite, 4.7% galena, 3.5% volynskite, 1.0% hessite, 0.2% chalcopyrite, with trace amounts of gold, michenerite, hawleyite (greenockite) and melonite.

Michenerite occurs as small 50-200μm subhedral to euhedral inclusions in altaite. Because of their greater hardness, these inclusions can generally be recognized by very slight relief differences developed during polishing. Grains are of distorted diamond shape to "kite" shape (Figs. 1, 2) or equant. In reflected light the mineral is pale pinkish purple, virtually indistinguishable from the associated volynskite. The absence of the cream or white tints referred to by Cabri et al. (1973) and Huang et al. (1974) is puzzling, but may be due to the fact that all our observations have been made on inclusions of michenerite in altaite. Although many grains are zoned from a core of testibiopalladite to a margin of michenerite, no color differences can be detected between these compositional zones, either in air or in oil immersion (Fig. 1).

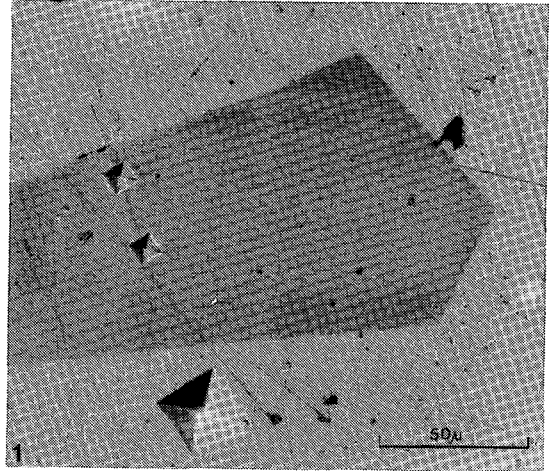Hardness measurements were made by indenting compositionally-different zones with 10

FIG. 1. Photomicrograph of euhedral michenerite–testibiopalladite in altaite. No evidence of zoning can be seen in the uniformly pale pinkish purple grain. Indentations with a 10g load show that the core of testibiopalladite is slightly harder (VHN 371) than the margin of michenerite (VHN 333). Both are significantly harder than altaite (VHN 50).

and 20g loads using a Vickers indenting objective; the resulting indentations are shown in Figure 1. The brittle nature of the mineral and the small grain size rendered heavier loads impracticable due both to fracturing of the grain and interference from neighboring grains. Determinations were calibrated using chalcopyrite
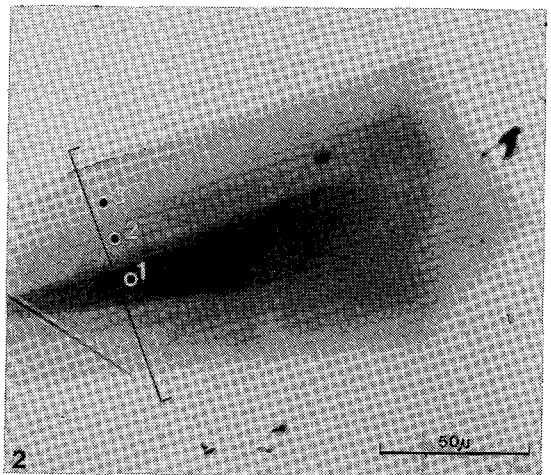

FIG. 2. Back-scattered electron image of the zoned michenerite–testibiopalladite shown in Fig. 1. The positions of the microprobe line-scan and the spot analyses are shown.

(VHN 195) and stainless steel (VHN 580). Both these standards gave VHN values that increased with increasing load. However, the results were nearly constant within the range 20–200g, with anomalously low but reproducible values for loads of 5 and 10g. Correction factors based on these standards were applied to the hardness determinations to give a value of VHN 371 for testibiopalladite (anal. 1, Table 2) and of 333 for michenerite (anal. 3, Table 2).

The value for michenerite is close to those found previously using 25g loads: 311–321 (Cabri *et al.* 1973), and 302 and 315 for platinian michenerites (Cabri & Laflamme 1976). They exceed, however, the value of 189 found for a platinian michenerite at a 10g load (Cabri & Laflamme 1976); this is consistent with our experience with the standards, as described above. Testibiopalladite determinations were performed at loads of 5 and 10g by P.M.M.R.G. (1974) and show the same tendency of low values, increasing with increasing load (Table 1). The greater hardness of testibiopalladite relative to michenerite, suggested by the comparable

determinations, at a 10g load, of 267 (P.M.M. R.G. 1974) and 189 (Cabri & Laflamme 1976), is confirmed in our study.

Evidence of compositional zoning in michenerite (Fig. 2) was first found in our samples by examining uncoated polished surfaces in a JSM–2 scanning electron microscope equipped with a wide-angle back-scattered electron detector (Robinson & Robinson 1978). Zones are concentric and show a progressive but somewhat steplike decrease in antimony and increase in bismuth from core to margin. Some reversals in zoning, whereby narrow, antimony-rich layers develop in the outer zone of michenerite, can be seen as dark (*i.e.*, low average atomic number) concentric bands in the back-scattered electron image (Fig. 2).

Grains that are completely enclosed in altaite are concentrically zoned. However, those that have a core of testibiopalladite at the contact of quartz and altaite have grown asymmetrically, giving a michenerite mantle on the three sides of the grain that are in contact with the altaite, and a continuous variation from michenerite
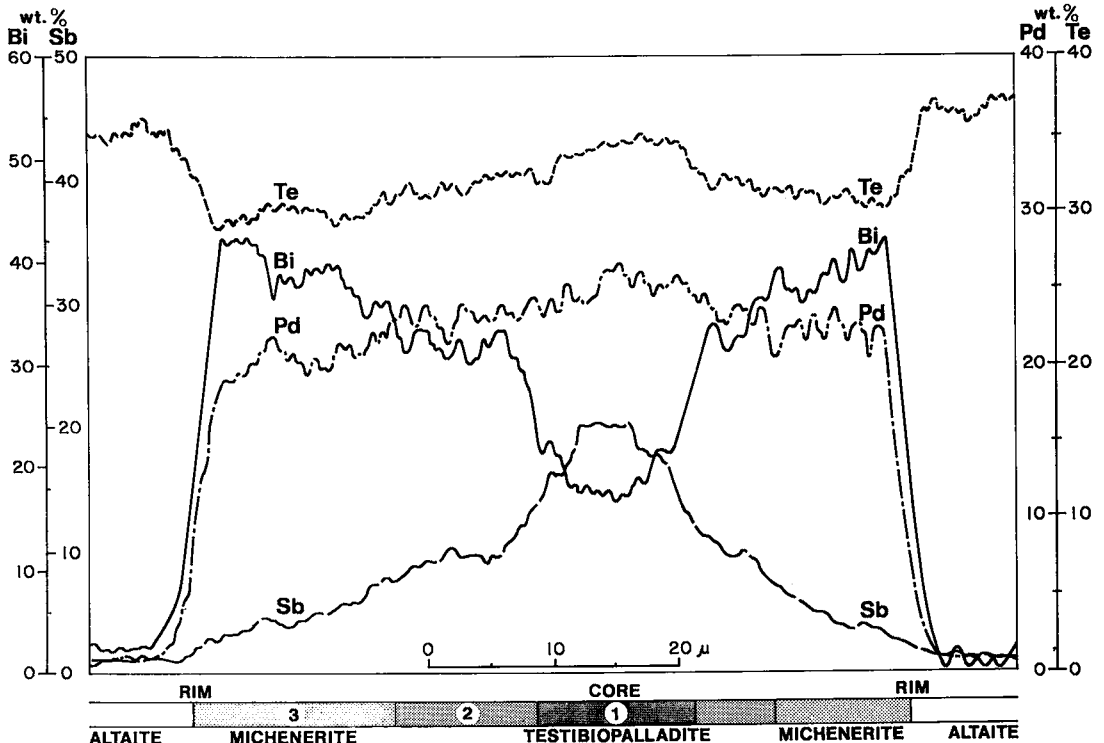

FIG. 3. Electron microprobe line-scan across the zoned michenerite–testibiopalladite. X-rays are generated from a spot approximately 5μ in diameter, so that element variation across zone boundaries is gradational relative to the sharp atomic-number contrast shown in Fig. 2.

through testibiopalladite to michenerite along the quartz interface. This suggests nucleation of the mineral after quartz crystallization. Whether growth was entirely from a hydrothermal fluid as seems likely from the zoning reversals or whether exsolution from altaite contributed to the outer bismuth-rich zones cannot be determined from existing data.

Scans across a zoned crystal (Fig. 3) were made using a Materials Analysis Company electron-probe microanalyzer operated at 19.3kV. Spot analyses from each of the three main compositional zones are given in Table 2. The microprobe analyses were made using crystal spectrometers LiF ($BiL\alpha$, $PtL\alpha$, $NiK\alpha$), RAP ($PbM\alpha$) and PET ($PdL\alpha$, $SbL\alpha$, $TeL\alpha$, $AgL\beta$); the standards were synthetic Bi_2Te_3(Bi,Te), synthetic PbTe(Pb), elemental Pt, Ni, Pd, Sb, Te and Ag, and corrections were made by the MAGIC IV program of Colby (1971).

Michenerite and testibiopalladite compositions have been calculated to formulae in a variety of ways. Cabri et al. (1973) and Cabri & Laflamme (1976) calculate (Pd+Pt+Ni) = 1; Genkin et al. (1972) and P.M.M.R.G. (1974) calculate (Bi+Sb+Te) = 2. In this paper, a compromise calculation based on a total of 3 atoms is adopted.

The core (analysis 1) has the composition of a bismuthian testibiopalladite $(Pd_{0.96}Pt_{0.02}Ni_{0.01})$-$_{\Sigma 0.99}(Sb_{0.74}Bi_{0.18}Ag_{0.01})_{\Sigma 0.93}Te_{1.08}$ in which there is slight substitution of Pt and Ni for Pd and some substitution of Te for Bi (or Sb). The analysis is similar to those reported from southwestern China (P.M.M.R.G. 1974) of $(Pd_{0.90}Ni_{0.06})_{\Sigma 0.96}$-$Sb_{0.64}Bi_{0.35})_{\Sigma 0.99}Te_{1.05}$ and $Pd_{0.94}(Sb_{0.67}Bi_{0.31})_{\Sigma 0.98}$-$Te_{1.08}$. Silver determinations were made using $AgL\beta$ to avoid the interference $PdL\beta$–$AgL\alpha$ pointed out by Cabri et al. (1973) as a possible explanation for the 1.8 wt.% Ag reported in michenerite by Genkin et al. (1972). Although count rates are low, values between 0 and 0.3 wt.% Ag were consistently determined, with an average value of 0.2 wt.% Ag. The silver has been arbitrarily assigned as substituting for (Sb, Bi). Lead was sought but fell below detection limits.

Analysis 2 is from the intermediate zone which has a composition $(Pd_{0.97}Pt_{0.02}Ni_{0.01})_{\Sigma 1.00}$-$(Bi_{0.56}Sb_{0.38}Ag_{0.01})_{\Sigma 0.95}Te_{1.06}$. It is similar in composition to the michenerite reported by Feather (1976) from Witwatersrand, $Pd_{1.05}(Bi_{0.55}Sb_{0.39})$-$Te_{1.01}$. Analysis 3 is from the marginal Bi-rich zone, with a composition similar to many reported michenerites.

Figure 4 shows the compositional fields of michenerites from Sudbury and testibiopalladites from China plotted in the triangular diagram Pd_2Te–Bi_2Te–Sb_2Te. The composition of zones
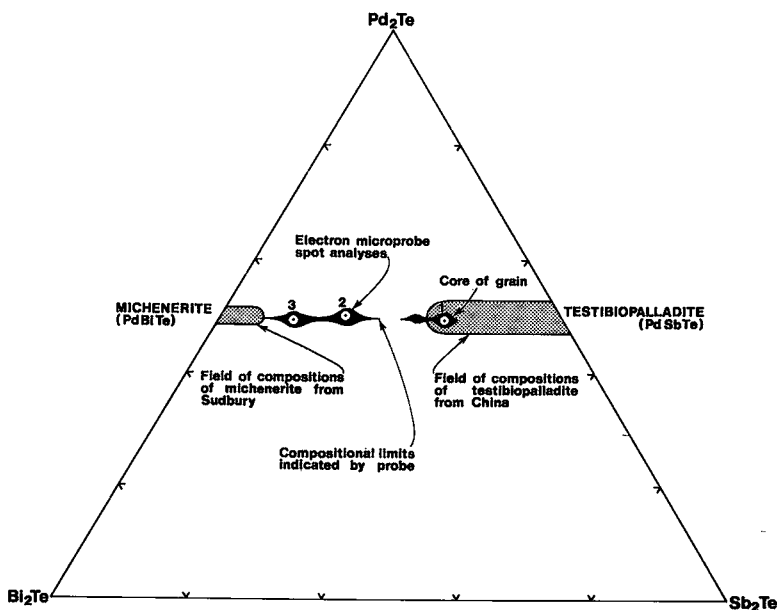

FIG. 4. Compositional limits, in atomic percent, of zoned michenerite–testibiopalladite from Kambalda, relative to michenerite from Sudbury (Cabri et al. 1973, Cabri & Laflamme 1976) and testibiopalladite from China (P.M.M.R.G. 1974). $Pd = \Sigma(Pd+Pt+Ni)$.

in the Kambalda michenerite–testibiopalladite have been deduced from the line scan and are controlled by spot analyses. Despite the apparent continuity of compositional variation in the X-ray scans, the sharp boundaries between the main compositional zones in the back-scattered electron image (Fig. 2) indicates that there is a step-like compositional variation between the core and margin and that not all possible compositions between michenerite and testibiopalladite were encountered in the study.

CONCLUSIONS

The first occurrence of testibiopalladite outside China is reported from Kambalda, Western Australia, where it occurs as cores to zoned inclusions of michenerite–testibiopalladite in altaite. A step-like, irregular increase in bismuth towards the margin of the zoned grains may reflect growth from hydrothermal solutions of changing composition or progressive exsolution from altaite. Experimental studies within the system Pd–Bi–Sb–Te are needed to clarify stability relationships between testibiopalladite and michenerite.

ACKNOWLEDGEMENTS

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