

## A NIOBIAN RUTILE – DISORDERED COLUMBITE INTERGROWTH FROM THE HURON CLAIM PEGMATITE, SOUTHEASTERN MANITOBA\*

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### ABSTRACT

A unique, irregular to quasi-graphic symplectite of 52.5 vol. % niobian rutile ( $(\text{Ti}_{1.42}\text{Nb}_{0.31}\text{Ta}_{0.04}\text{Fe}_{0.26}\text{Mn}_{0.01}\text{Sn}_{0.01})_{\Sigma 2.04}\text{O}_4$ ) and 47.5 vol. % disordered titanian columbite ( $(\text{Ti}_{0.32}\text{Nb}_{2.37}\text{Ta}_{0.06}\text{Fe}_{1.02}\text{Mn}_{0.22}\text{Sn}_{0.01})_{\Sigma 4.00}\text{O}_8$ ) occurs in the Huron Claim pegmatite, Manitoba. The intergrowth is found locally with larger grains of a single-phase but compositionally inhomogeneous niobian rutile with an average formula  $(\text{Ti}_{0.72}\text{Nb}_{0.75}\text{Ta}_{0.03}\text{Fe}_{0.56}\text{Mn}_{0.01}\text{Sn}_{0.01})_{\Sigma 2.08}\text{O}_4$ . Both types of niobian rutile conform to the general characteristics of this rutile variety, with  $(\text{Fe}+\text{Mn})/(\text{Nb}+\text{Ta}) > 0.5$ ,  $\text{Fe} > \text{Mn}$ ,  $\Sigma$  cations  $> 0.5$  oxygens, and have the monorutile structure. Both rutile phases have lower Nb/Ta and higher Fe/Mn than the titanian columbite, which is also the case for rutile-columbite pairs and coexisting tapiolite-columbite from other localities. The bulk composition of the symplectite does not match the composition of the single-phase niobian rutile grains, and the symplectite differs from exsolution textures typical of other occurrences. The intergrowth seems to be the product of an initial precipitation of extremely Fe,Nb-rich rutile, followed by simultaneous symplectitic crystallization of disordered titanian columbite and Fe,Nb-poor rutile. The intergrowth was probably quenched after crystallization or shielded from volatile agents, as most compositional analogues of all three phases from other localities are exsolved.

**Keywords:** rutile, columbite, symplectite, granitic pegmatite, Manitoba.

### SOMMAIRE

On signale un exemple unique de croissance symplectitique irrégulière à quasi-graphique de rutile niobifère [52.5 % par volume,  $(\text{Ti}_{1.42}\text{Nb}_{0.31}\text{Ta}_{0.04}\text{Fe}_{0.26}\text{Mn}_{0.01}\text{Sn}_{0.01})_{\Sigma 2.04}\text{O}_4$ ] et de columbite titanifère désordonnée [47.5 %,  $(\text{Ti}_{0.32}\text{Nb}_{2.37}\text{Ta}_{0.06}\text{Fe}_{1.02}\text{Mn}_{0.22}\text{Sn}_{0.01})_{\Sigma 4.00}\text{O}_8$ ] dans la pegmatite Huron Claim (Manitoba). L'intercroissance est associée localement à des cristaux monophasés, plus gros et hétérogènes de rutile niobifère de composition moyenne  $(\text{Ti}_{0.72}\text{Nb}_{0.75}\text{Ta}_{0.03}$

$\text{Fe}_{0.56}\text{Mn}_{0.01}\text{Sn}_{0.01})_{\Sigma 2.08}\text{O}_4$ . Les deux types de rutile niobifère se conforment aux caractéristiques générales de cette variété de rutile:  $(\text{Fe}+\text{Mn})/(\text{Nb}+\text{Ta}) > 0.5$ ,  $\text{Fe} > \text{Mn}$ ,  $\Sigma$  cations  $> 0.5$  atomes d'oxygène, structure du monorutile. Les deux possèdent un rapport Nb/Ta inférieur et un rapport Fe/Mn supérieur à la columbite titanifère, comme les associations rutile-columbite et tapiolite-columbite rencontrées ailleurs. La composition globale de la symplectite ne correspond pas à celle des cristaux de rutile niobifère monophasé; de plus, l'intercroissance ne ressemble pas aux exemples d'exsolution typiques d'autres localités. La texture résulterait de la cristallisation initiale d'un rutile fortement enrichi en Fe et Nb, suivie par la cristallisation simultanée symplectitique de la columbite titanifère désordonnée et d'un rutile plus pauvre en Fe et Nb. L'intercroissance aurait été brusquement refroidie ou protégée des effets de la phase volatile après sa cristallisation, car dans la plupart des autres localités, ces trois phases sont exsolvées.

(Traduit par la Rédaction)

**Mots-clés:** rutile, columbite, symplectite, pegmatite granitique, Manitoba.

### INTRODUCTION

A suite of specimens from the Huron Claim pegmatite was collected in the early 1930s by T.L. Walker and subsequently deposited in the collection of the Department of Mineralogy and Geology, Royal Ontario Museum, Toronto. During a recent study of the pegmatite (Paul, in prep.), we found that one of Walker's specimens (M17303A) contains a peculiar black opaque nodule, which proved to be an intergrowth of niobian rutile and disordered titanian columbite. The scarcity of niobian rutile occurrences in Canada (Traill 1970, 1974, Petruk 1972, Černý & Turnock 1971), the unique symplectitic structure predominant in the intergrowth and the high Fe,Nb content of its rutile component prompted the detailed study reported in this paper.

### OCCURRENCE

The Huron Claim pegmatite is a member of

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the Shatford Lake pegmatite group in the Winnipeg River pegmatite district of southeastern Manitoba (Černý *et al.* 1981), in the English River subprovince of the Archean Superior Province of the Canadian Shield. The first radiometric age in excess of 2 Ga ever recorded was established on uraninite from this pegmatite (DeLury & Ellsworth 1931, Ellsworth 1932). Mineral paragenesis of the Huron Claim pegmatite encompasses 34 species, with a diversified assemblage of Be, Nb-Ta, Ti, REE, Y, Zr, Th and U minerals (Walker 1931, Ellsworth 1932, Černý & Bristol 1972, Paul, in prep.).

The sample M17303A consists mainly of albitic plagioclase with subordinate and partly leached quartz; columbite, a brown euxenite-like phase and pale green zircon are scattered throughout. The intergrowth described here is roughly ellipsoidal and 2 x 3 cm in size on the exposed surface.

## THE INTERGROWTH

### *Phase composition and structure*

The intergrowth consists of two textural components: (1) finely symplectitic niobian rutile and columbite, which overwhelmingly predominates over (2) local coarser patches of niobian rutile (Fig. 1a, b). Rounded crystals of zircon are scattered throughout and are commonly surrounded by radial cracks (Fig. 1c). Aggregates of a metamict euxenite-like phase with a characteristic mud-crack fracture pattern generally occur along the margins of the intergrowth, exhibiting smooth convoluted contacts (Fig. 1d).

The niobian rutile - columbite symplectite has an average composition of 52.5 vol. % niobian rutile and 47.5 vol. % columbite. "Grains" of each species are usually mutually interconnected in an intricate vermiform or dactylic manner, with no clear distinction be-

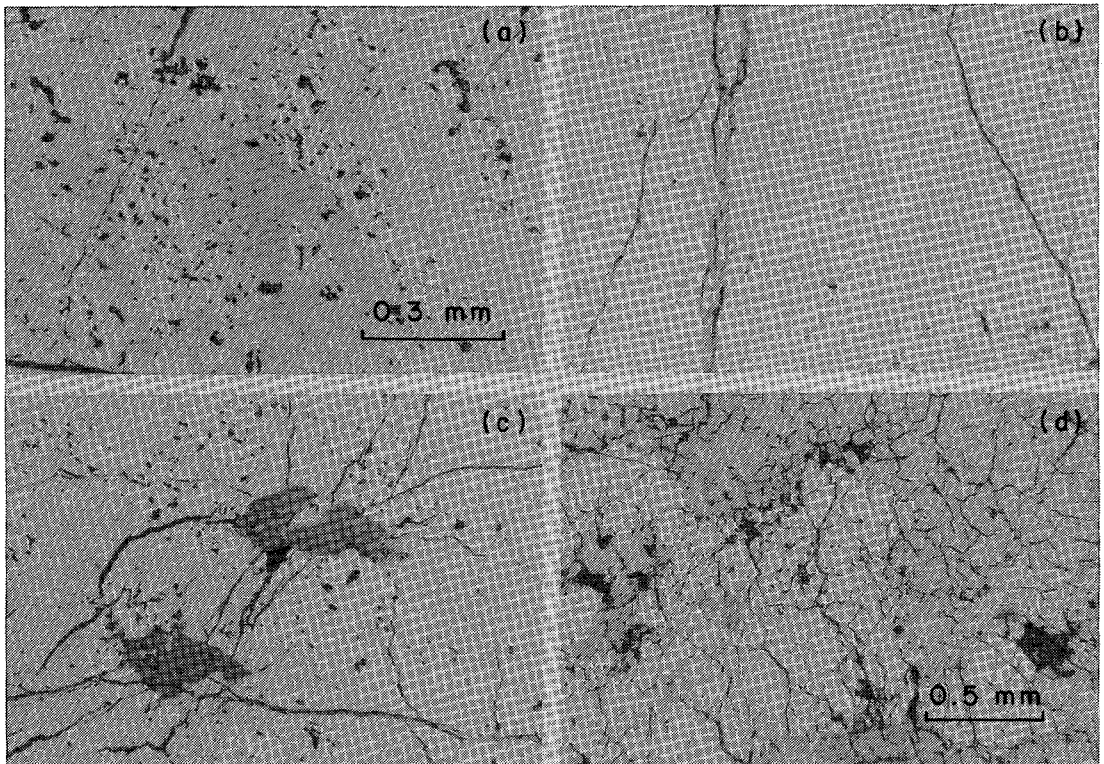


FIG. 1. (a) Fine grained symplectite of niobian rutile (light) and columbite (dark), with ragged grain boundaries; (b) quasi-graphic coarse grained symplectite of niobian rutile (light) and columbite (dark), with simple angular grain boundaries (scale as in a); (c) inclusions of zircon (dark) in the symplectite, surrounded by radial fractures (scale as in a); (d) the symplectite (light area) surrounded by an euxenite-like mineral (dark area with mud-crack fissure pattern).

tween host and inclusion. The "grain" size of both minerals varies between 0.1 and 0.5 mm. The finer grained symplectite is usually irregular to ragged, whereas the coarser parts tend to be more quasi-graphic (Fig. 1a, b). Both "grain" size and structure show random variation and gradual transitions.

The coarser patches of niobian rutile have an irregular equidimensional shape and vary between 1 and 2 mm. They are either sharply separated from both phases of the surrounding symplectite or transitional into its niobian rutile component.

### Physical properties

In transmitted light, both species of the intergrowth are faintly translucent along the edges of thin splinters. Niobian rutile is strongly dichroic in dark brown ( $\epsilon$ ) and dark green ( $\omega$ ), whereas columbite has a relatively weak pleochroism in reddish dark brown tones. In reflected light, both minerals are cream colored, columbite being darker and slightly brownish. Coarse patches of niobian rutile have slightly higher reflectance than the same species intimately intergrown with columbite. Both birefractance and anisotropic effects between

crossed polars are more pronounced in columbite than in niobian rutile.

X-ray powder-diffraction data show niobian rutile to have the monorutile structure and columbite to be a disordered phase with an ixiolite-type unit cell. Heating at 1000°C for 16 hours in air converts the disordered columbite into an ordered phase, indicating identity with the "pseudo-ixiolite" of Nickel *et al.* (1963). Refinement of unit-cell dimensions from X-ray powder-diffraction data calibrated with  $\text{CaF}_2$  ( $a$  5.4602 Å) gives an average of  $a$  4.637(2),  $c$  3.002(3) Å for niobian rutile,  $a$  4.723(2),  $b$  5.709(2),  $c$  5.116(2) Å for natural disordered columbite and  $a$  14.273(3),  $b$  5.724(2),  $c$  5.059(2) Å for the ordered columbite obtained after heating.

### Chemical composition

Electron-microprobe analyses were performed on the MAC 5 instrument in the wavelength-dispersion mode at 15 kV with a specimen current of 0.04 A, using the following standards: titanite (for Ca, Ti),  $\text{Ba}_2\text{NaNb}_5\text{O}_{15}(\text{Nb})$ , mangantantalite (Mn, Ta), chromite (Al, Fe, Mg) and cassiterite (Sn). The EMPADR VII program (Rucklidge & Gasparini 1969) was used

TABLE 1. CHEMICAL COMPOSITION OF NIOBIAN RUTILE AND DISORDERED COLUMBITE

	1	2	3	4	5	6	7	8	9	10
$\text{Ta}_2\text{O}_5$	3.4	3.1	3.4	4.5	3.5	2.9	2.7	2.9	2.9	3.6
$\text{Nb}_2\text{O}_5$	46.9	47.7	20.7	24.5	19.3	70.9	70.6	70.2	70.5	68.6
$\text{TiO}_2$	27.5	27.3	65.9	57.2	66.5	4.8	6.7	5.7	5.8	7.1
$\text{SnO}_2$	0.5	0.7	0.9	1.1	0.8	0.2	0.3	0.1	0.3	0.6
$\text{Al}_2\text{O}_3$	0.4	-	0.1	0.2	0.4	0.05	0.3	0.3	-	-
$\text{FeO}^*$	20.0	19.2	9.1	11.5	9.3	17.3	15.8	16.4	15.9	18.5
MnO	0.3	0.5	0.01	0.1	-	3.9	2.9	3.5	3.6	1.3
MgO	0.3	0.1	-	0.1	0.1	0.2	0.2	-	0.1	0.1
CaO	0.02	0.01	-	0.04	0.01	0.1	0.1	0.03	0.02	0.1
	99.32	98.61	100.11	99.24	99.91	100.35	99.60	99.13	99.12	99.9
Ta	0.03	0.03	0.03	0.04	0.03	0.06	0.06	0.06	0.06	0.07
Nb	0.74	0.76	0.28	0.35	0.26	2.38	2.36	2.37	2.38	2.30
Ti	0.72	0.72	1.49	1.35	1.50	0.27	0.37	0.32	0.33	0.39
Sn	0.01	0.01	0.01	0.014	0.01	0.006	0.01	0.003	0.01	0.017
Al	0.015	-	0.004	0.01	0.01	0.004	0.02	0.02	-	-
$\text{Fe}^{2+}$	0.58	0.56	0.23	0.30	0.23	1.07	0.97	1.02	0.99	1.15
Mn	0.01	0.015	-	0.002	-	0.25	0.18	0.22	0.23	0.08
Mg	0.02	0.0003	-	0.005	0.003	0.02	0.02	-	0.013	0.007
Ca	-	-	-	-	-	0.006	0.01	0.002	0.001	0.008
	2.125	2.098	2.044	2.071	2.073	4.066	4.00	4.015	4.014	4.022
$\text{Ta}/(\text{Ta}+\text{Nb})$	0.04	0.04	0.10	0.10	0.10	0.03	0.03	0.03	0.03	0.03
$\text{Mn}/(\text{Mn}+\text{Fe})$	0.02	0.03	0.00	0.01	0.00	0.19	0.16	0.18	0.19	0.07
$\frac{(\text{Fe}+\text{Mn}+\text{Mg})}{(\text{Nb}+\text{Ta})}$	0.79	0.73	0.74	0.79	0.80	0.55	0.48	0.51	0.51	0.52

\* Total Fe calculated as FeO. Electron-microprobe analyses by R. Chapman and F.C. Hawthorne. 1. Large grain of Fe,Nb-rich niobian rutile adjacent to #8. 2. Large grain of Fe,Nb-rich niobian rutile adjacent to #4 and #10. 3. Niobian rutile in symplectite, adjacent to #7. 4. Niobian rutile in symplectite, adjacent to #2 and #10. 5. Niobian rutile in symplectite, adjacent to #9. 6. Disordered columbite in symplectite. 7. Disordered columbite in symplectite, adjacent to #3. 8. Disordered columbite in symplectite, adjacent to #1. 9. Disordered columbite in symplectite, adjacent to #5. 10. Disordered columbite in symplectite, adjacent to #2 and #4.

to reduce the data. Three adjacent niobian rutile and columbite pairs from the fine symplectite, two grains of the coarse niobian rutile and two additional columbite grains were analyzed quantitatively. Table 1 presents the data cross-referenced to the analyses of adjacent phases.

Columbite compositions are relatively uni-

form, irrespective of their association, with very high Fe/Mn and Nb/Ta ratios and relatively high Ti contents. In contrast, niobian rutile shows a remarkable difference between the symplectite and the coarse patches. In association with columbite in the symplectite, niobian rutile is relatively poor in Fe and Nb and, as also verified by energy-dispersion spectra of

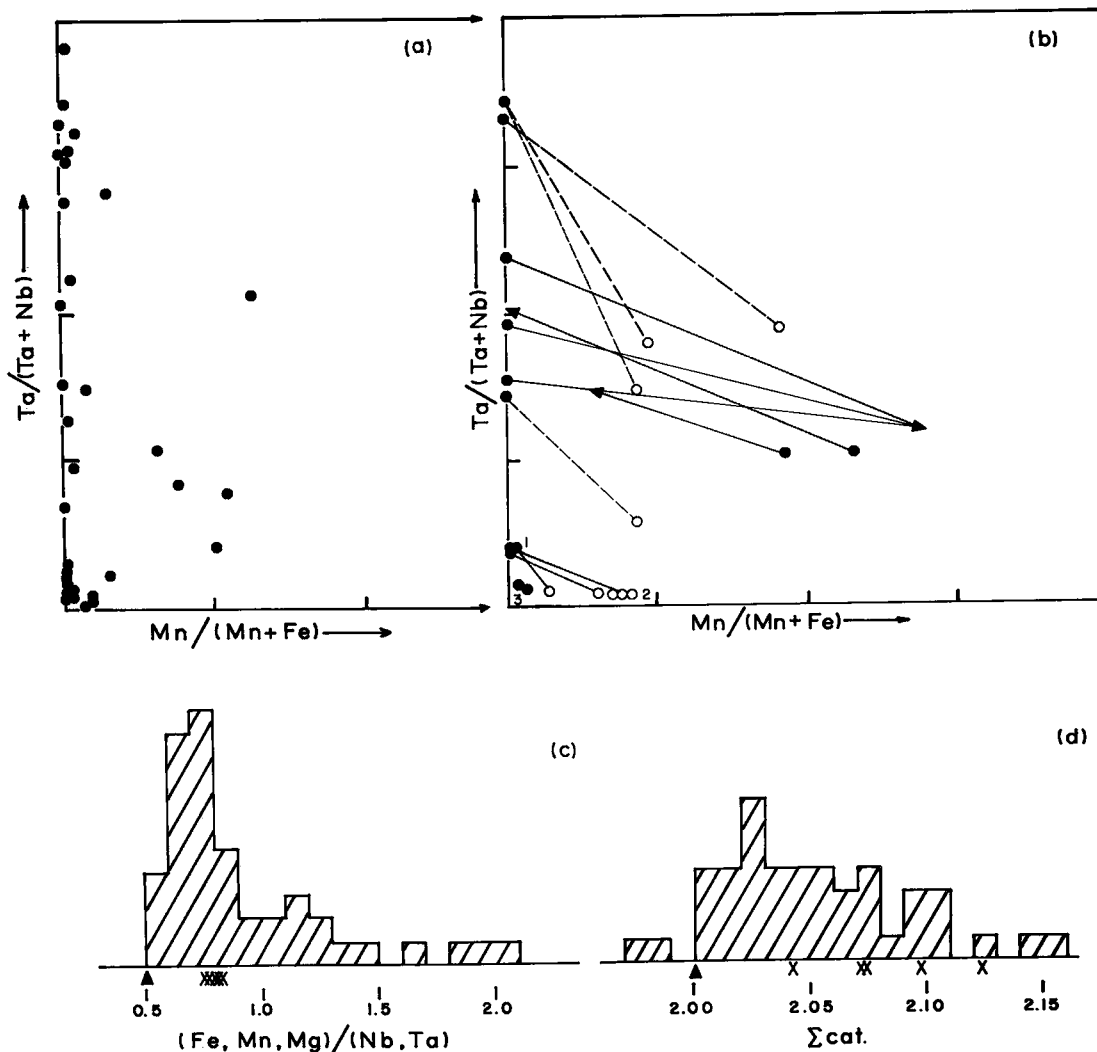


FIG. 2. (a) Bulk compositions of niobian and tantalian rutiles in terms of atomic  $Ta/(Ta+Nb)$  and  $Mn/(Mn+Fe)$  ratios from the literature; (b) coexisting and exsolved niobian-tantalum rutile + columbite-tantalite pairs in the above plot: 1 niobian rutile and 2 columbite of the Huron Claim symplectite, 3 large grains of the Huron Claim niobian rutile; for other points from the literature, solid dot is the host and arrowhead is the inclusion/exsolution; solid and open circle pairs are coexisting discrete grains of rutile and columbite phases, respectively; (c) frequencies of the  $(Fe, Mn, Mg)/(Nb, Ta)$  ratio in niobian and tantalian rutile (at.); triangle denotes the "ideal" value, X marks the Huron Claim ratios; (d) cation totals in niobian and tantalian rutile, based on 4 oxygens (symbols as in c). See text for sources of data.

additional spots, rather uniform in composition. On the other hand, the coarse grains of niobian rutile are rich in Fe and Nb and are inhomogeneous. Linear scans of the coarse grains re-

veal considerable variation in all three major elements; Ti distribution is reciprocal to that of Nb, which is in turn only slightly correlated with Fe, indicating a variable Fe/Nb ratio.

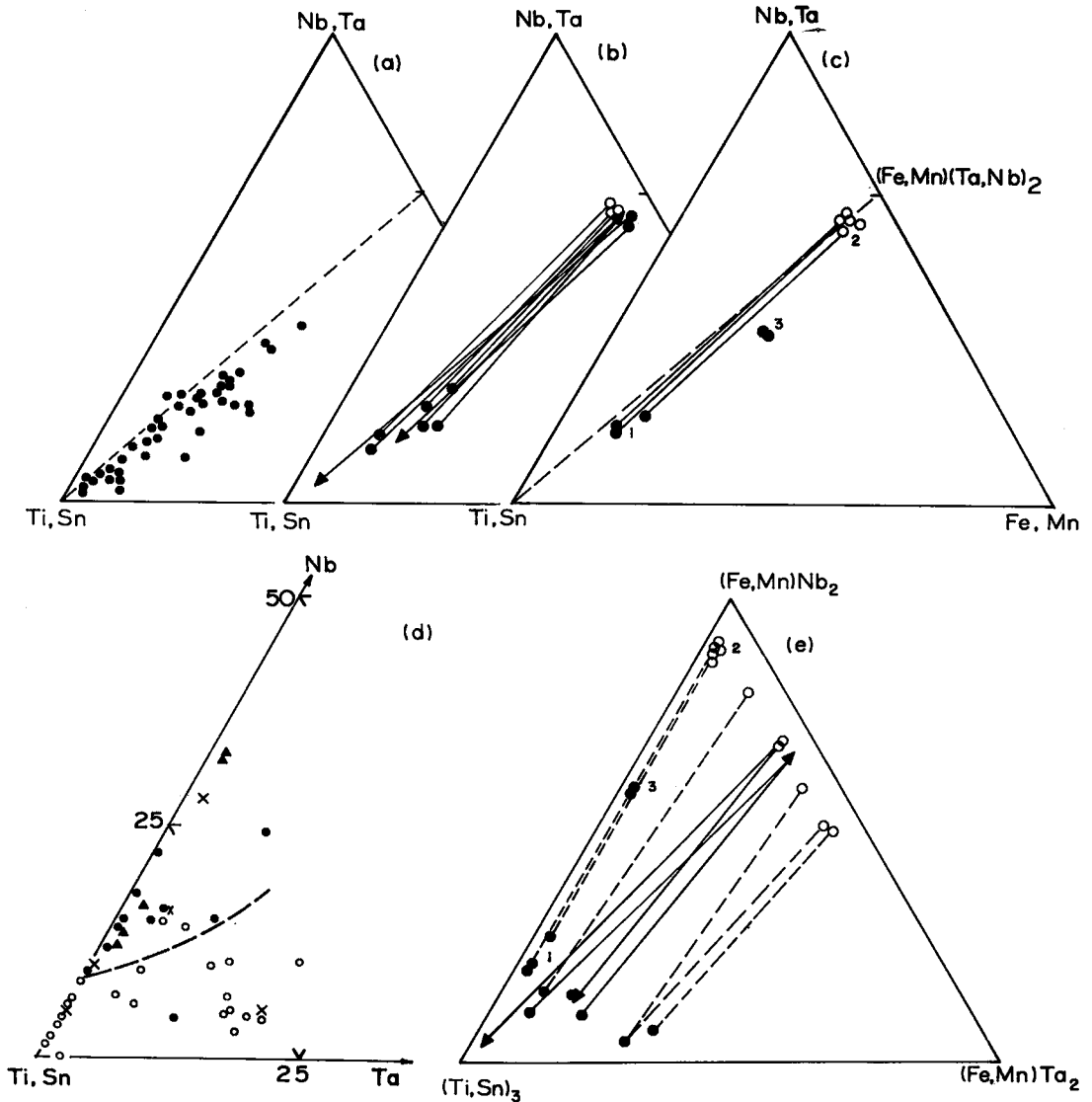


FIG. 3. (a,b,c) Niobian and tantalian rutile compositions in the (Ti,Sn)-(Fe,Mn)-(Nb,Ta) triangular plot (at. %); (a) bulk compositions plot largely on the (Fe,Mn)-rich side of the (Ti,Sn)-"columbite" ("tapiolite") join; (b) coexisting and exsolved niobian and tantalian rutile + columbite-tantalite pairs (symbols as in Fig. 2b); (c) niobian rutile (solid dots) and columbite (open circles) from Huron Claim (numbering as in Fig. 2b); (d) bulk compositions of niobian and tantalian rutile in the (Ti,Sn)-Nb-Ta triangular plot (at. %); open circles: homogeneous phases, X: homogeneity unknown, solid dots: exsolved phases that are, with a single exception, Nb-rich (above the dashed line), triangles: Huron Claim compositions; (e) coexisting and exsolved niobian-tantalian rutile + columbite-tantalite in the (Ti,Sn)<sub>3</sub>-(Fe,Mn)Nb<sub>2</sub>-(Fe,Mn)Ta<sub>2</sub> triangular plot (at. %; symbols as in Figure 2b). See text for sources of data.

Despite the differences between the two types of niobian rutile, all compositions of this phase have Fe/Mn and Nb/Ta ratios lower than the columbite.

## DISCUSSION

### *Niobian rutile*

New data on niobian and tantalian rutile published during the last 17 years have not substantially modified the general characteristics of these minerals established by Černý *et al.* (1964). The data summarized by these authors (except those replaced by better analyses), compositions given by Chukhrov (1960), Kornetova (1963), Distler *et al.* (1963), Lima de Faria & Quadrado (1966), Feldman & Konopleva (1969), Lugovskoi & Stolyarova (1969), Siivola (1970), Kornetova *et al.* (1971), Černý & Turnock (1971), Arnaudov & Petrusenko (1972), Clark (1974), Černý *et al.* (1979, 1981), Desborough *et al.* (1980) and the analyses collected by Kornetova (in Chukhrov & Bondshedt-Kupletskaya 1965) confirm the following features of niobian and tantalian rutile:

(1) They are highly variable in Nb/Ta but invariably show a high Fe/Mn ratio, which is also the case for the tetragonal tapiolite  $\text{FeTa}_2\text{O}_6$  (Fig. 2a).

(2) Their composition is commonly described as a solid solution of  $\text{TiO}_2$  with a tapiolite-type phase ( $\text{Fe} \gg \text{Mn}$ ) ( $\text{Ta} \geq \text{Nb}$ ) $_2\text{O}_6$ , but they contain ( $\text{Fe} \gg \text{Mn} + \text{Mg} + \text{Ca}$ ) in excess of the ideal  $(\text{Fe} + \text{Mn})/(\text{Nb} + \text{Ta})$  ratio of 0.50 (Figs. 2c, 3a). This may be caused by the incorporation of either the rutile-type compound  $\text{Fe}^{3+}\text{NbO}_4$ , or Fe alone independent of the substitutions involving (Nb, Ta).

(3) Cation totals normalized to 4 oxygens exceed the ideal value of 2.00 (Fig. 2d). The reasons for this may range from oxygen deficiency to  $\text{Ti}^{4+} \leftrightarrow 2\text{Fe}^{2+}$  or  $3\text{Ti}^{4+} \leftrightarrow 4\text{Fe}^{3+}$  substitutions (or both).

(4) Many specimens contain a high proportion of  $\text{Fe}^{3+}$ , to the exclusion of  $\text{Fe}^{2+}$  (Gordiyenko & Kulchitskaya 1962, Lugovskoi & Stolyarova 1969).

(5) Most phases with compositions showing appreciable substitution and  $\text{Nb} > \text{Ta}$  are inhomogeneous, containing exsolved columbite or, more rarely, ilmenite (Fig. 3d). The only exsolved mineral with  $\text{Nb} < \text{Ta}$  is the  $\text{Fe}^{3+}$ -rich sample described by Lugovskoi & Stolyarova (1969). The preference of Nb for the orthorhombic columbite structure is well known

from compositions of coexisting columbite-tapiolite pairs.

(6) All minerals examined to date have monorutile unit cells. Trirutile and birutile X-ray-diffraction patterns reported by Flinter (1959) were found to represent monorutile + columbite and monorutile + ilmenite mixtures, respectively (Černý *et al.* 1964, Flinter 1964).

Owing to the intimate intergrowth with columbite, the oxidation state of Fe in the Huron Claim niobian rutile could not be determined. In other respects, this mineral closely matches the above characteristics (Figs. 2b, c, d and 3c, e). The only exception appears to be the lack of unambiguous exsolution textures (as discussed below). This is rather surprising, as both types of this niobian rutile are rich in Nb and Fe, and the large grains of this phase (Table 1, anal. 1, 2) surpass in this respect all other analyzed samples (Fig. 3b, d, e).

### *Disordered columbite*

The disordered columbite phase is relatively close in composition to end-member ferrocolumbite in terms of Fe/Mn and Nb/Ta ratios, but is rich in Ti (Table 1, anal. 6–10). However, the Ti content does not attain the extreme values of 8.8–12.8 wt. %  $\text{TiO}_2$  known in disordered columbites from other localities (Sveshnikova *et al.* 1965, Černý *et al.* 1981).

### *Cation partitioning between niobian rutile and columbite*

Considering the abundance of niobian rutile with exsolved columbite phase (Fig. 3d) and several occurrences of columbite with niobian rutile inclusions that also seem to be of exsolution origin, data on the composition of these mineral pairs are very scarce. Figures 2b, 3b and 3e show the compositions reported by Siivola (1970) and Černý *et al.* (1981), complemented by several analyses of coexisting columbites-tantalites and niobian-tantalian rutiles from Černý *et al.* (1979, 1981).

All these compositions indicate preferred partitioning of Fe and Ta into the rutile phase and enrichment of columbite in Mn and Nb, regardless of the diverse genetic relationships of the two minerals. This corresponds to the compositional differences between Ti-poor columbite-tantalite and tapiolite as documented, for example, by Foord (1976) and Clark & Fejer (1978). The columbites show only limited variation in Ti, in contrast to the much more extensive and variable ( $\text{Fe} \gg \text{Mn}$ ) and ( $\text{Nb}, \text{Ta}$ )

substitution in the rutile phase (Fig. 3e). These relationships suggest that the compositions could follow an asymmetric solvus skewed toward the columbite side in the system  $TiO_2$ - $FeNb_2O_6$ . The absence of exsolution in Ta-rich compositions indicates either a great reduction of the two-phase region in the system  $TiO_2$ - $FeTa_2O_6$  or a more persistent metastability of homogeneous phases in this system, or both.

The compositions of the two phases constituting the Huron Claim intergrowth conform to the general partitioning pattern outlined above. Despite the overall Fe,Nb predominance in both columbite and rutile, the Fe/Mn and Nb/Ta ratios indicate the preference of Fe and Ta for the rutile structure. However, the large grains of the Nb,Fe-rich rutile phase seem to have been metastable during crystallization of the enclosing symplectite.

#### *Origin of the intergrowth*

The relatively constant proportions and composition of the two phases constituting the symplectite and its widespread quasi-graphic structure do not favor a metasomatic origin. Exsolution is common in niobian rutiles (Fig. 3d), but the rutile host predominates over the exsolved grains or lamellae of columbite in such cases, and consequently the texture is different from that observed in the Huron Claim specimen (Černý *et al.* 1964, Plates 9 to 16). If the symplectite were of exsolution origin, the large grains of extremely Fe, Nb-enriched rutile would represent a metastable anomaly in an aggregate of exsolved phases; the large grains of niobian rutile could not represent relics of a homogeneous parent phase, as their composition is different from the bulk chemistry of the symplectite (Fig. 3c).

The textural and compositional features of the symplectite suggest that it formed by simultaneous crystallization of both phases, following a short period of precipitation of the large Fe, Nb-rich grains. After crystallization, the whole intergrowth must have been thermally quenched or shielded against late fluids and volatile compounds that could promote re-equilibration, as most niobian rutile phases with original compositions in the compositional range of the symplectite-forming and large-grain rutile phases have exsolved (Fig. 3d).

#### CONCLUSIONS

The data from the literature, together with the present results, define distinct composi-

tional characteristics for niobian and tantalian rutile, and they suggest regular patterns in its compositional relationship with associated columbite-tantalite minerals. However, there are insufficient experimental results to explain all aspects of the crystal chemistry, and microprobe data on compositions of coexisting phases are scarce. Both problems are currently under study by the present authors; detailed examination of new finds of niobian and tantalian rutile is desirable.

#### ACKNOWLEDGEMENTS

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