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Electron-Probe Microanalysis of Light Elements in Multiphase Diffusion Couples*

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Abstract. Wavelength-dispersive electron-probe microanalysis of transition metal carbide and nitride diffusion couples was investigated as a tool to quantitatively determine portions of phase diagrams. In cross-sections, diffusion couples generally show a sequence of several phases composed of crystallites with different orientations and hardness values. Thus, the polishing procedure can adversely affect the accuracy of the analysis due to creation of an uneven surface (relief effect). In addition, as a result of the polishing process the edges of the samples are rounded so that the concentration at the original sample surface is difficult to measure. The most rapid and straightforward procedure to detect influences of the relief effect is a plot of the non-normalized analytical total vs. distance, even where the concentration profile is discontinuous at the interface between phase bands. It is shown that careful experimental preparation, as well as the application of restricted diffusion geometry, has several advantages for establishing phase diagrams.

Key words: Carbide, nitride, diffusion couple, relief effect, diffusion geometry.

Multiphase reaction diffusion couples have proved successful in both the evaluation of phase equilibria and in the measurement of layer growth rates, especially in binary systems. The sequence of phases in the metallographically prepared cross-section of a diffusion couple represents a portion of the phase diagram of the corresponding system [1, 2]. This portion can cover a complete isothermal section (compositional range from one pure component to the other for a given temperature) or, if the diffusion couple is annealed in a temperature gradient, it can even cover a temperature range. While the metallographic observation gives a qualitative image of the sequence and number of phases occurring in the system, compositional information is available whenever microanalytical techniques can be successfully applied to measure the local composition quantitatively. By far the most powerful technique for this procedure is wavelength-dispersive electron-probe microanalysis (WDS-EPMA).

The present paper describes some advances achieved in our laboratories by applying this technique to diffusion couples of transition metal–carbon and nitrogen systems. In this study, the group V transition metal carbides and nitrides are emphasized. The technique was also applied to Ti-N [3], Hf-N [4], Cr-N and Cr-C [5] diffusion couples.

Experimental

Preparation of Diffusion Couples

The diffusion couples were either prepared by reacting metals at temperatures up to 2200 °C in a high-purity nitrogen atmosphere (nitrides) or by pack carburization of metals in a graphite bed (carbides). Details of the preparation techniques are described elsewhere [6]. Restricted sample geometry was applied (plane-sheet and wedge-type) which leads to enhancement of the width of phase bands as compared with quasi-infinite geometry [7].

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Metallographic Preparation

The samples were cut with a diamond disc perpendicular to the diffusion layers and embedded in cold-setting resin (Araldite® (Bayer) or, for electrically conductive embedding, in Technovit 4000® (Kulzer-Heraeus)) and ground with a 20 µm diamond disk. After this procedure a 3-µm diamond paste was applied on a hard cloth (Struers DP-NAP) for polishing. In order to obtain optimal surface conditions so as to be able to distinguish phase bands in polarized light, an aqueous SiO_2 suspension (Struers OPS) was applied, which provides a combination of mechanical and chemical attack. This procedure yields excellent microstructural images but creates an uneven and topographically structured surface (relief) which, as will be shown, is unfavorable for EPMA measurements.

For an alternative route for sample preparation a special lapping technique was applied by Radiometer GmbH (Struers/Logitech), Germany. This procedure is usually required when surface roughness and flatness must be on the order of a few tenths of a µm (e.g. for piezoelectric crystals). For the present purpose the samples were lapped with an abrasive on a cast iron disc with water as a lubricant. SiC powder (mesh 600) was used in the first stage and then aluminium oxide (3 µm) as an abrasive. The polishing time was 50 min. After that procedure Syton polishing fluid was applied with a polyurethane disc for 20 min. The formation of a surface relief could not be completely avoided, but the lapping procedure, which could be further optimized, resulted even so in a much flatter surface than with standard metallographic polishing.

Preparation and Characterization of Standards

The careful characterization of the standards used for calibration of the radiation yield is most important. Many carbides and nitrides have a homogeneity range and dissolve impurities such as oxygen, nitrogen and carbon, so that it may be somewhat incorrect to take the nominal composition of a phase, e.g. 'Cr_2N', as the actual composition 33.3 at % N. Throughout all the investigations performed we generally applied 'homotypic standardization' [3]. This means that, for each system, standards were applied containing the same number and type of components as the samples to be investigated. This is a straightforward approach to guarantee the nearest possible correspondence between standard and sample on an experimental basis in order to offset any effects, such as peculiar background shapes, line coincidences and bonding shifts, that cannot be compensated satisfactorily by physical models and/or numerical algorithms.

The standards were prepared by diffusion of nitrogen or carbon into compact metal until complete equilibration was obtained (hot-pressing of carbide and nitride powders resulted in samples of much poorer quality). This could be achieved by applying high temperatures in conjunction with thin starting metal sheets. The composition was measured by chemical analysis (Dumas-GC [8]) and weight gain. Table 1 gives the data for the carbide standards and shows a close agreement between weight gain (for VN the composition was calculated from the lattice parameter) and chemical analysis.

EPMA Measurements

WDS-EPMA was performed with a Cameca SX50 microprobe equipped with five spectrometers and a variety of crystals to make accessible the characteristic radiations of the full range of elements (atomic weight greater than He). For light elements W/Si multilayer crystals were applied, since they show a much better radiation yield for the N-$K\alpha$ line and a less complicated background shape for any of the light elements than ordinary crystals like Pb stearate. A variety of different conditions was chosen for the beam current and voltage, for operating the oxygen jet and for the counting time per step; these are listed in Table 2. The conditions were optimized taking also into account the necessary boundary condition that long line-scans consisting of several hundred points have to be performed across the diffusion couples in order to be able to keep the total investigation time to an acceptable level (e.g. counting times of more than 30 s per step were not applied due to this precondition). Most of the recommendations of Bastin and Heijligers [9, 10] for the proper choice of experimental measurement conditions such as application of an oxygen jet, cold trap, background measurement, beam current and voltage have been found to be applicable in the present study. The oxygen jet was used only for carbon analysis. The oxygen pressure in the chamber was adjusted so that the carbon line intensity could be kept at a minimum. Since decontamination needed a few seconds, it proved to be advantageous to decontaminate the surface of the measurement position prior to each step. This was performed by introducing a delay of 6 s for beginning the counting after the sample holder had moved to the new position. Although some compounds are sensitive to oxidation and the oxygen signal increases with time, no perceptible carbon loss could be detected within the applied counting time. This is consistent with the observations of Bastin and Heijligers [9].

The counting time was chosen in such a way that the confidence level was around 95% (depending on the phase analysed, see below) using the formula

$$\varepsilon(\%) = \frac{2 * \sqrt{(P+B) * t}}{(P-B) * t} * 100 \qquad (1)$$

Table 1. Analytical results for standards of the group V transition metal carbides and nitrides where the composition was measured from both weight gain and chemical analysis (Dumas GC)

Phase	Sample thickness (µm)	Annealing temperature (°C)	Annealing time (h)	Composition by Dumas GC (at% C or N)	Composition by weight gain (at% C or N)
δ–VC_{1-x}	290	1500	90	17.34	17.27
δ–NbC_{1-x}	300	1830	50	11.24	11.29
δ–TaC_{1-x}	240	2000	44	6.17	6.23
δ–VN_{1-x}	800	1700	170	48.41	48.85[a]
δ–NbN_{1-x}	330	1700	96	46.85	46.90
ε–TaN	120	1710	180	7.13	7.13

[a] From the linear relationship lattice parameters vs. [N]/[V] ratio.

Table 2. Parameters for the analysis of carbide and nitride diffusion couples adjusted on homotypic standards and measured counting rates. All measurements with cold trap. Oxygen flow rate adjusted so that the oxygen pressure in the chamber was 3×10^{-4} mbar

Element	Beam conditions (kV/nA	Crystal[a]	Peak position ($10^5 \sin \Theta$)	+ bg[b] ($10^5 \sin \Theta$)	− bg[b] ($10^5 \sin \Theta$)	Counting time (s)	Counting rate (c/s nA)
C	10/100	PC1	75418	7000	7000	15	5.3
V	10/100	LIF	62179	600	–	15	118.3
C	8/300	PC1	75353	7000	7000	30	1.6
Nb	8/300	PET	65435	700	–	10	63.8
C	6/300	PC1	75291	5400	5400	30	1.5
Ta	6/300	TAP	28222	700	–	6	158.7
N	10/100	PC1	54101	13000	–	10	21.6
V	10/100	LIF	62179	600	–	10	111.5
N	6/200	PC1	52873	3840	3840	15	3.6
Nb	6/200	PET	65421	2200	1440	15	23.3
N	10/300	PC1	54086	9000	4000	30	3.5
Ta	10/300	TAP	28227	600	–	10	340.2

[a] PC1: 59.8 Å, LiF (200): 4.026 Å, PET (002): 8.742 Å, TAP (100): 25.9 Å
[b] Background position to be added to (+ bg) or subtracted from (− bg) the peak position.

Where P is the peak intensity, B is the background intensity and t is the counting time.

Results and Discussion

Confidence Levels

Data calculated from Eq. (1) with $\varepsilon = 2.5$ for the carbide and nitride phases investigated are given in Table 3. From these values it can be seen that the necessary counting time to reach the 95% confidence level is highest for the niobium carbide phases. For β–Nb_2C the necessary precision can only be reached at a counting time of 49 s. Since the counting time had to be kept constant during automatic line scans, the level of confidence for some phases is lower and for some phases better than 95%. Furthermore it is obvious that the metal phases representing solid solutions of small amounts of carbon or nitrogen (V(C), Nb(C), Ta(C), Nb(N)) cannot be analysed with comparable confidence levels within acceptable counting times. For carbon analysis, this is additionally affected by the contribution of carbon originating from contamination. The latter cannot be completely avoided and thus even small contributions of carbon contamination give a substantial contribution to the measured carbon content. For nitrogen analysis in the solid solutions of V(N) and Ta(N) the situation is better, since these metals dissolve a high amount of nitrogen if diffusion couples with thick layers of nitride phases are prepared. For example, in the case of Nb-C (maximum carbon content about 1 at%) the necessary counting

Table 3. Values (round) for the counting rates at the peak maximum (P) of carbon or nitrogen peaks and in the background (B), see Eq. 1, for several carbide and nitride phases studied in this work. Counting rates refer to phases near their highest carbon and nitrogen content, respectively. Beam conditions, see Table 2

Phase	Peak (counts/s)	Background (counts/s)	Counting time (s)
α–V(C)	40	23	1395
β–V_2C	380	39	23
ζ–V_4C_{3-x}	419	39	20
δ–VC_{1-x}	576	39	14
α–Nb(C)	70	62	13200
β–Nb_2C	271	62	49
ζ–Nb_4C_{3-x}	298	61	41
δ–NbC_{1-x}	401	58	25
α–Ta(C)	34	29	16128
β–Ta_2C	288	33	32
ζ–Ta_4C_{3-x}	368	38	24
δ–TaC_{1-x}	416	40	21
α–V(N)	400	15	18
β–V_2N	1300	16	5
δ–VN_{1-x}	2180	12	3
α–Nb(N)	112	102	13700
β–Nb_2N	550	170	32
δ–NbN_{1-x}	700	110	15
α–Ta(N)	314	214	338
β–Ta_2N	757	204	20
δ–TaN_{1-x}	1076	210	11
ε–TaN	1251	205	9

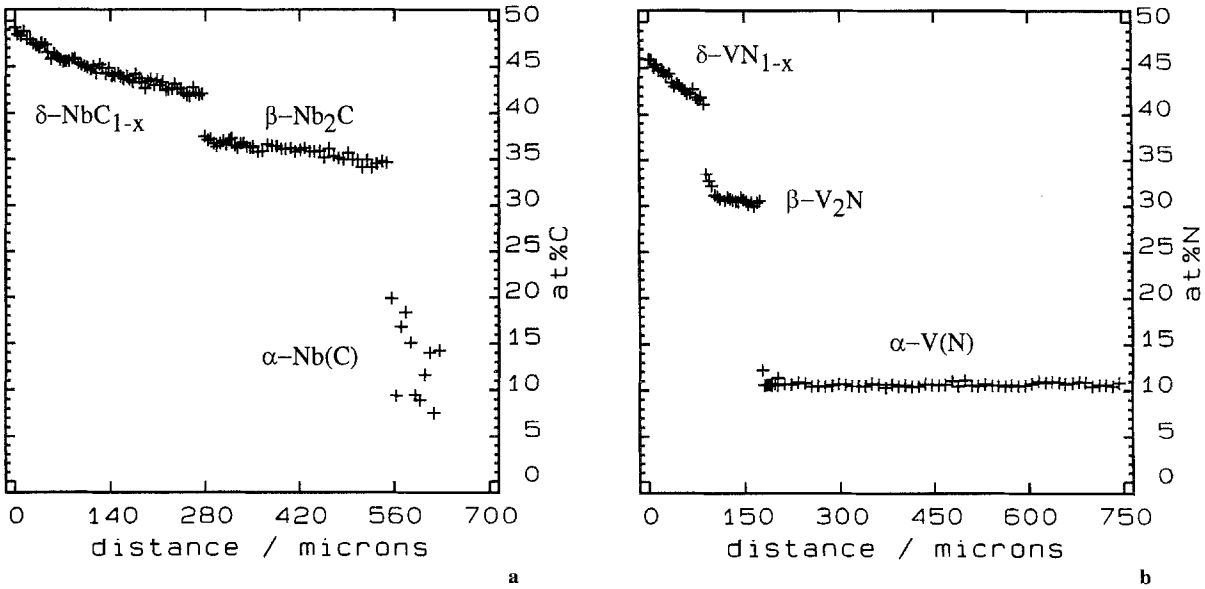

Fig. 1. EPMA line scans on **a** Nb-C (left) and **b** V-N (right) diffusion couples. The carbon content in α–Nb(C) shows a large scatter due to the impossibility of applying sufficient measurement time (Table 3) and the influence of remaining carbon contamination, whereas in α–V(N) the nitrogen signal is high and can be measured without scatter. Smooth diffusion profiles are observed in all carbide and nitride phases

time for a carbon analysis in α–Nb(C) under the given conditions would be very high (13200 s), Table 3, whereas the necessary counting time for the nitrogen analysis in α–V(N) (maximum nitrogen content about 12 at%, depending on the temperature) is 18 s. The situation is reflected in the comparison of the line scans of a Nb-C with a V-N diffusion couple (Fig. 1) – the data for α–Nb(C) are scattered whereas a smooth profile was obtained for α–V(N).

In addition, it should be noted that, apart from larger subcarbide and subnitride precipitates occurring at grain boundaris, finely dispersed particles of these phases can be present in the metal matrix. The latter do not cause observable scatter in the non-metal profile but shift the measured contents to higher than the true values.

Influence of Surface Topography

In order to make observable the diffusion-band structure it is often convenient to use polarized light which enhances the color contrast between neighboring phases. For this end it is necessary to obtain excellently polished specimens. One way to obtain this is the combination of low load and long polishing times. In addition, the use of chemically active SiO_2 suspension as a polishing fluid is very favorable in the very last polishing step. However, this polishing procedure pro-

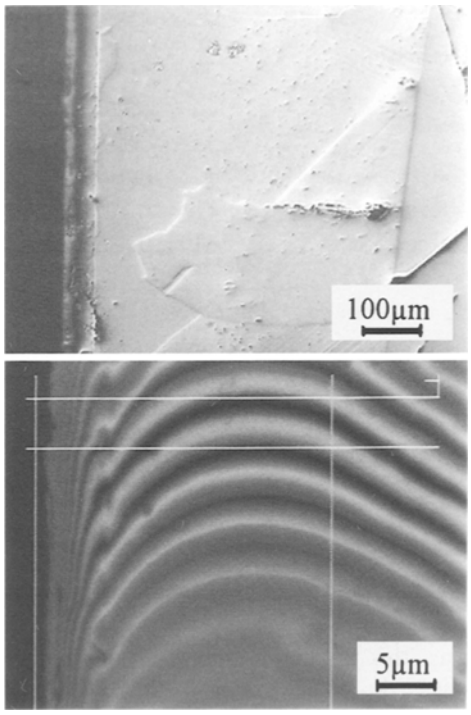

Fig. 2. Top: Microstructure of an Hf-N diffusion couple which shows grain boundaries of differently oriented grains due to relief polishing (dark regions: pores and cracks). Bottom: Interference microscope pattern from the edge of the diffusion couple showing the rounding of the edge upon polishing. The distance between two neighbouring stripes is 0.3 μm (the white lines are for distance measurement purposes)

duces a topographically structured surface (relief) which is unfavorable for EPMA. To illustrate this effect, a diffusion couple (Hf-N) is shown in Fig. 2. The microstructure (top) shows that the grains are visible due to relief polishing. At the bottom an interference microscope image of the outermost edge of this couple is given, showing the rounding of the edge upon polishing (the difference between two stripes corresponds to ca. 0.3 μm). If EPMA scans on a such polished diffusion couples are performed, untypical non-metal diffusion profiles are obtained. Differences in the topography of the polished surface (height differences) cause differences in the excited volume and differences in the emitted X-ray radiation yield as a function of beam position on the sample. The most rapid and straightforward test for relief effects is a plot of the analytical total vs. distance, which is shown in the lower plot of Fig. 3. The analytical total is defined as the non-normalized sum of all

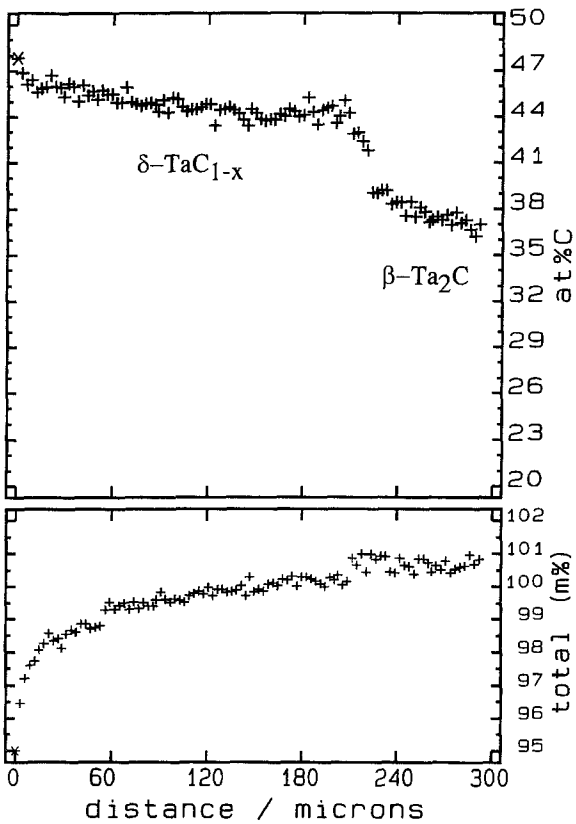

Fig. 3. Microprobe scan on a Ta-C diffusion couple prepared by standard metallographic polishing. Top: The carbon profile in δ–TaC_{1-x} is too flat for the type of the diffusion process that had been performed (in-diffusion of carbon leading to a surface concentration of about 50 at% C). Bottom: Plot of the analytical total (in mass%) vs. distance, indicating that the measurement is influenced by relief effects (radiation loss at the sample edge and a discontinuity at the δ/β interface)

independently measured constituents of the sample. In case of correct quantification the analytical total should lie between 99 and 101% and any deviation is a direct indication of the local variation of total X-ray yield. The lower plot shows a substantial deviation to low total values because of the rounding of the edge of the specimen. It was found that the detected characteristic radiation from the metals is more influenced by the surface topology than is the radiation of non-metals. This is additionally affected by the relative position of sample and detectors.

By use of the analytical total, relief effects are also detected if they occur at the interfaces of adjacent phase bands, where compositional discontinuities are present representative of the two-phase region of the phase diagram. If discontinuities in the analytical total occur at positions of the interfaces, such as in Fig. 3 at the δ–TaC_{1-x}/β–Ta_2C boundary, the measurement of the width of the two-phase field may not be correct.

In order to minimize these relief effects, no SiO_2 polishing step (or only a short one) should be applied. Then, however, the visibility of diffusion bands in the light-optical microscope can be unsatisfactory, especially in the microprobe which generally is equipped with optical microscopes of lower quality than metallographic microscopes. The best way to avoid relief effects is the use of lapping techniques that have been developed to arrive at extremely even and plane surfaces for crystals in piezoelectric devices and lasers. Figure 4 shows EPMA results of a Ta-C diffusion couple prepared with standard metallographic techniques (left) and with lapping techniques (right). On the left discontinuities in the bottom plot of the analytical total vs. distance can be seen (indicated by arrows). For the lapped specimens such discontinuities are absent or much less pronounced, showing the better performance of the lapping procedure for metallographic polishing. Note that the concentration range shown for the Ta-C system (Figs. 3 and 4) is within about 2 m% C only.

Influence of Specimen Geometries

At the edge of the specimens the difficulties of preparation are even more severe than for internal interfaces. Due to poor edge retention in general the outermost region of a diffusion couple cannot be analysed. In the case of a very steep concentration profile a step scan over this region does not yield the true composition of the metalloid-rich phase boundary of the outermost

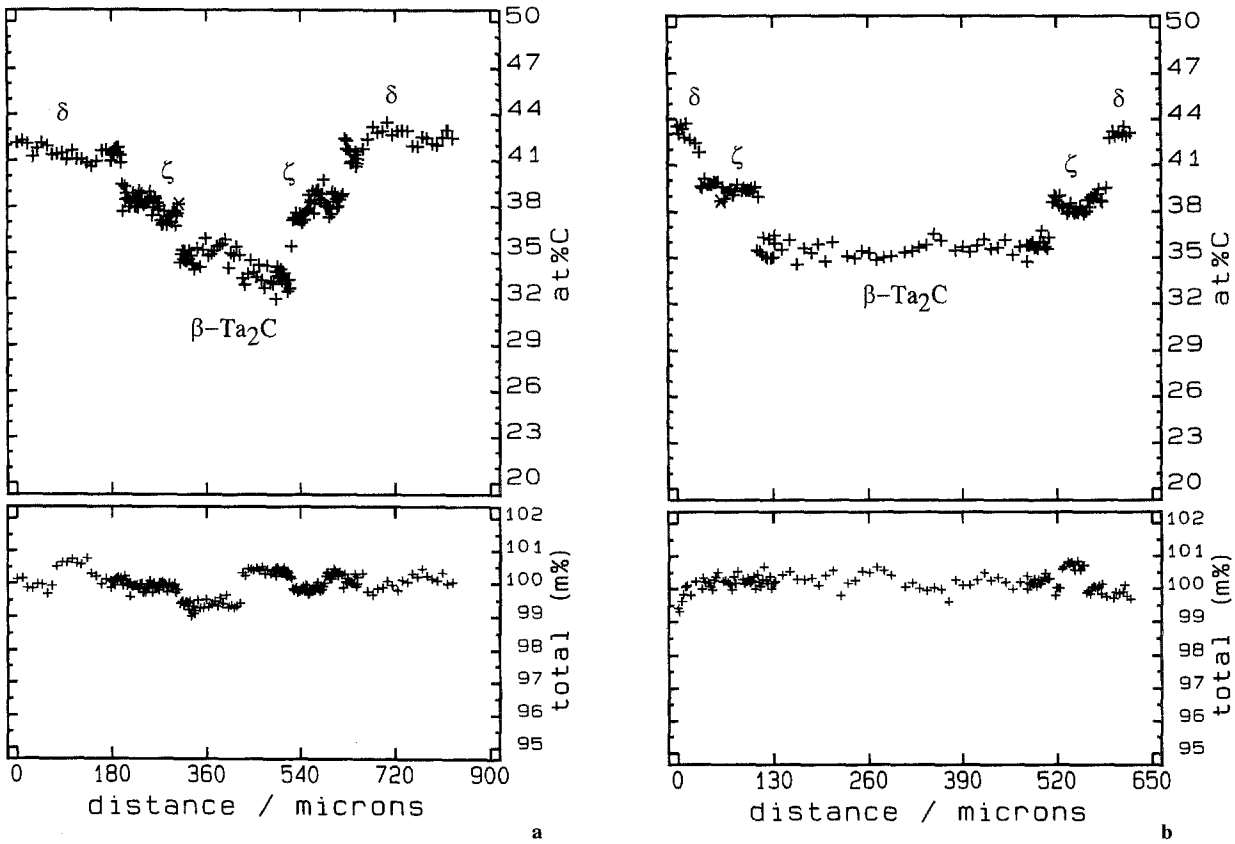

Fig. 4. EPMA scans together with the analytical total (in mass%) across the core of Ta-C specimens that had been metallographically prepared (**a**) under standard conditions (left) and (**b**) by lapping (right). Note the discontinuities in the analytical total due to relief polishing. Lapping produces a much flatter specimen surface and hence the surface topography has less influence on X-ray radiation yield

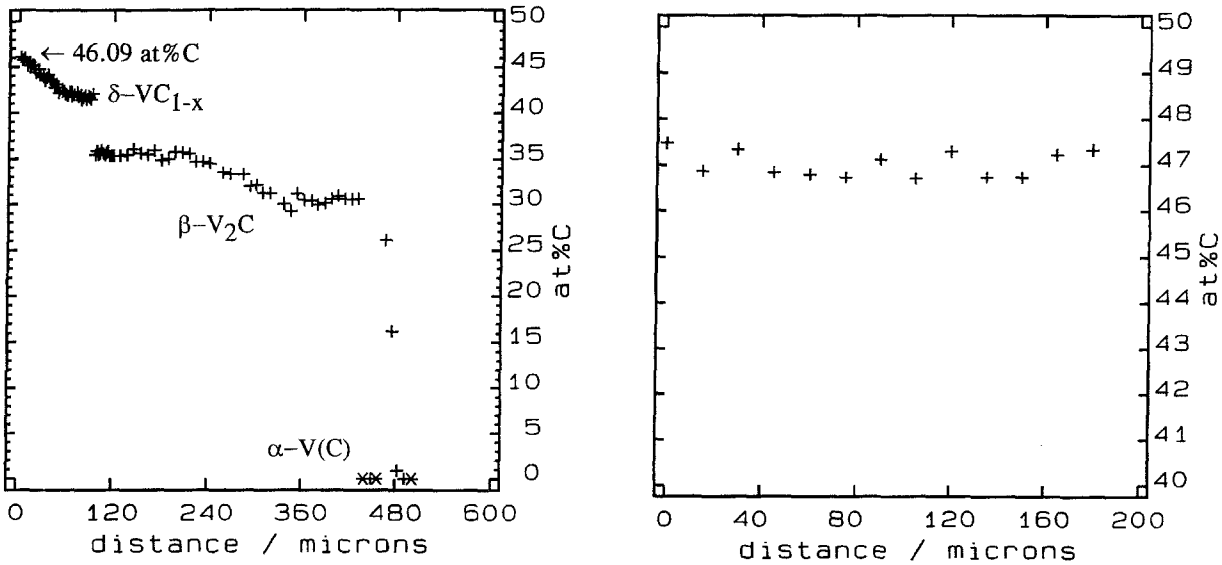

Fig. 5. Left: EPMA scan across one half of a V-C diffusion couple. The surface concentration (46.09 at% C) of δ–VC_{1-x} is too low because of the rounding of the edge during specimen preparation. Right: The tip of the wedge-type sample shows a horizontal carbon profile from which the carbon-rich composition of δ–VC_{1-x} could be determined (47.09 at% C)

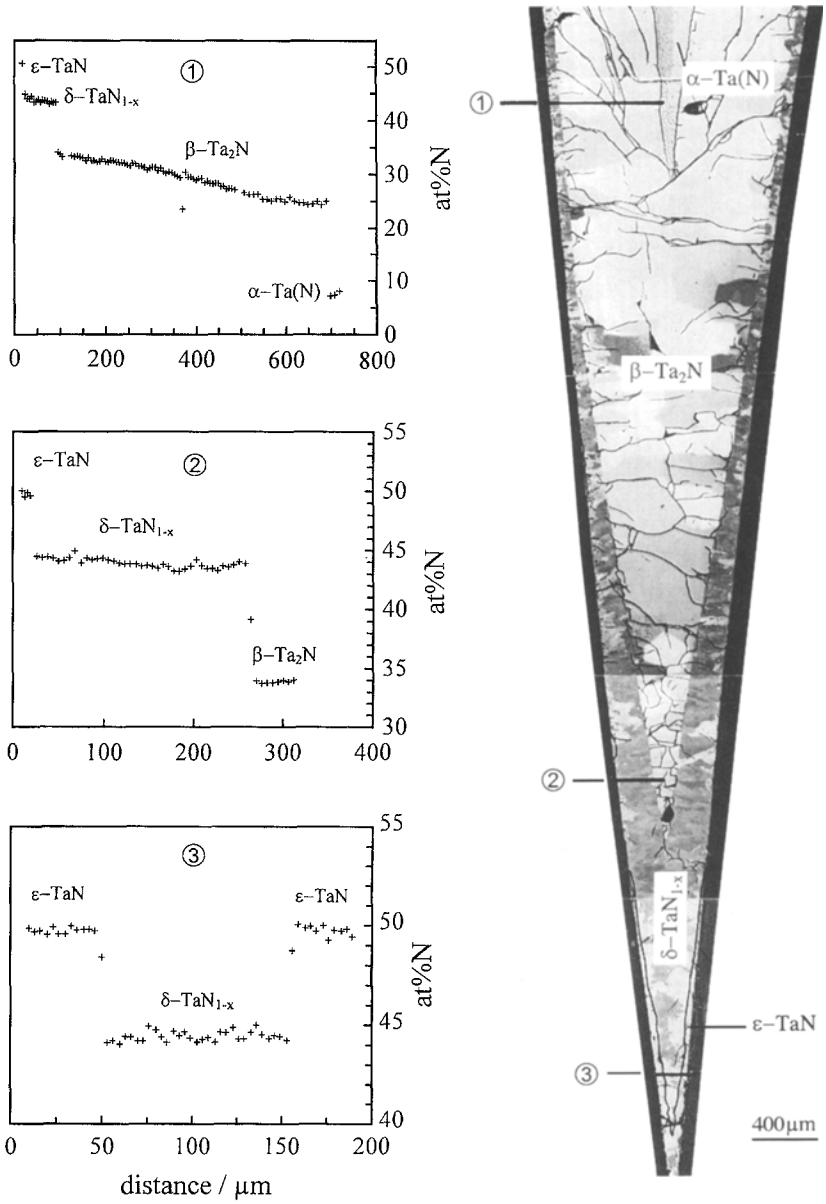

Fig. 6. Microstructure of a wedge-type Ta-N diffusion couple and the corresponding EPMA nitrogen profiles across the couple. The data points for the line compounds ε–TaN and δ–TaN_{1-x} could be recorded due to the phase band enlargement as a result of restricted diffusion geometry (scan No. 2 and 3). The ε/δ interface at the bottom of the microstructure is emphasized. The colour microstructure of this sample is accessible under the URL: http://info.tuwien.ac.at/physmet/tan2.html

phase. Such a situation was discussed by Bastin and Heijligers [11], where the composition at the edge of a Ti-N diffusion couple annealed at 1400°C in flowing N_2 (pressure about 1 bar) was measured to be 45 at% N even when the position of the sample towards the detector was favorable (by turning the sample by 180 deg). The radiation loss because of the rounding – in connection with the presence of a very steep diffusion profile at the outermost region due to the presumably strongly concentration-dependent diffusion coefficient – led to the misleading conclusion that the TiN phase is sub-stoichiometric in equilibrium with the gas phase at the applied annealing conditions.

Since the rounding of the edge can never be completely avoided, an alternative procedure for the measurement of the homogeneity region of the outer phase in a diffusion couple is the preparation of wedge-type diffusion couples. The homogeneity regions of all the internal phases can be measured in the same way as for plane-sheet couples but the composition of the outermost phase is accessible by measuring the tip of the wedge-type diffusion couple. At the wedge tip the diffusion profile is practically horizontal as compared to any other position, so the rounding effect does not perceptibly influence the measurement. Corresponding results are shown in Fig. 5 for the V-C system.

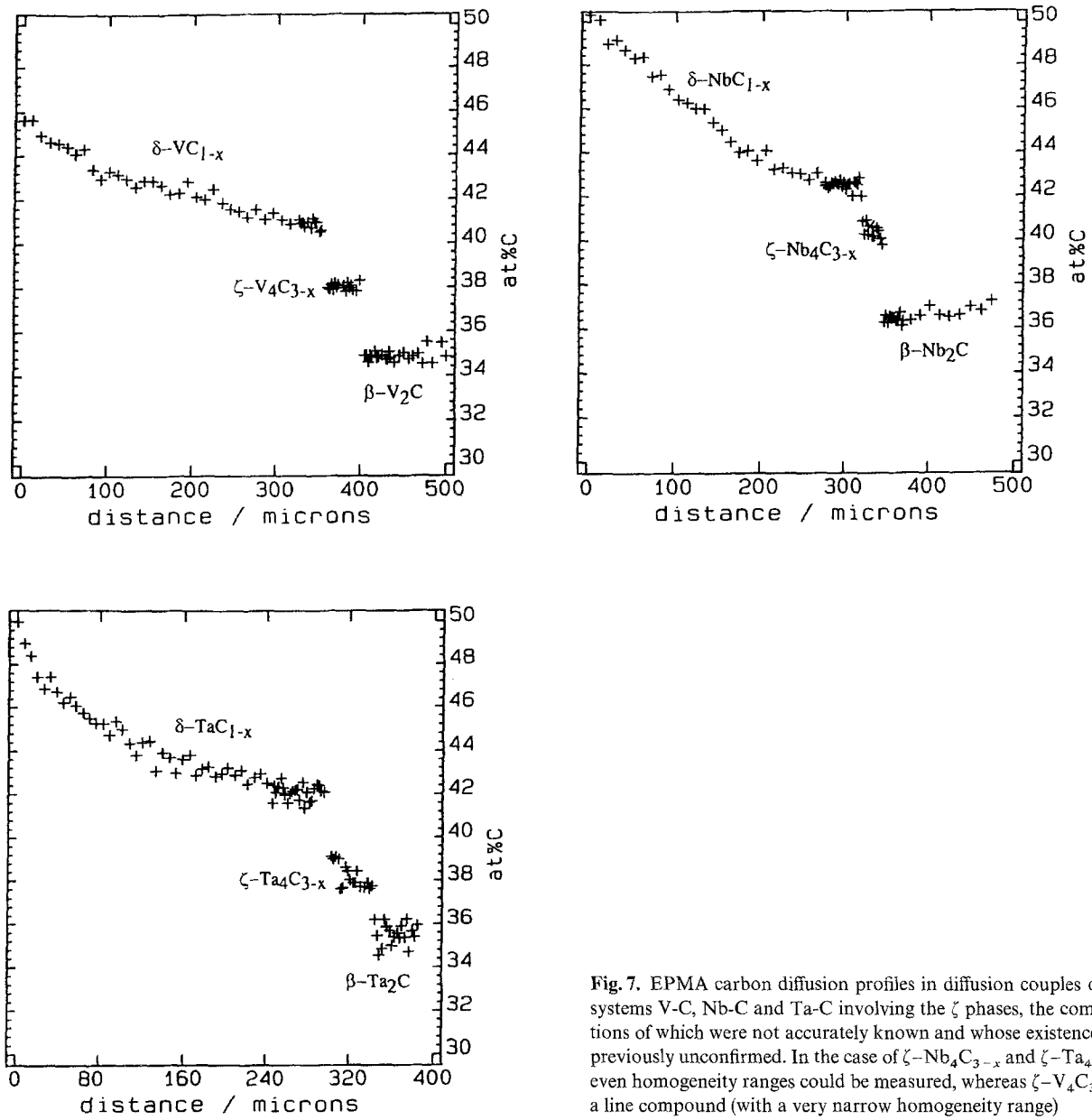

Fig. 7. EPMA carbon diffusion profiles in diffusion couples of the systems V-C, Nb-C and Ta-C involving the ζ phases, the compositions of which were not accurately known and whose existence was previously unconfirmed. In the case of ζ-Nb_4C_{3-x} and ζ-Ta_4C_{3-x} even homogeneity ranges could be measured, whereas ζ-V_4C_{3-x} is a line compound (with a very narrow homogeneity range)

A measurement of the cross-section gives the composition of all phases at internal phase boundaries but yields only 46.10 and 46.08 at% C at the opposite edges for the carbon-rich composition of δ-VC_{1-x}, whereas measurement at the tip yielded 47.09 ± 0.08 at% C (from 2 line scans), in excellent agreement with the literature. Several other carbide and nitride diffusion couples were measured by this procedure and the composition at the carbon-rich or nitrogen-rich phase boundary of the monocarbide or mononitride, respectively, could be established.

The lateral resolution of EPMA is, depending on the measurement conditions, about 1–2 μm. This is sufficient for the measurement of the average concentration of diffusion bands with a width of only 5–10 μm, but insufficient to yield any diffusion profiles within these bands. In order to make thin diffusion bands accessible to diffusion profile measurements, which then yield the homogeneity range of the phase, the specimens must either be annealed for long periods or thin or wedge-type specimens should be used. In thin as well as in wedge-type specimens the influence of plane-sheet

(restricted) diffusion geometry is such that the phase bands grow faster than in the semi-infinite case [4]. The growth rate can be expressed by a time-dependent parabolic rate constant which is the greater, the smaller the thickness of the sample [12]. Because of this, the phase bands in a wedge-type sample become broader, the smaller the sample thickness. This effect can be made use of for the measurement of the composition or homogeneity region of phases which otherwise would form very thin diffusion bands. Such measurements were performed for a variety of systems. A scan for a Ta-N couple is shown in Fig. 6. The outermost phase in this couple is ε–TaN and forms usually a very thin phase band which is rounded during polishing and is not accessible to statistically reliable analysis. By use of thin or wedge-type samples this phase band was broadened so that several data points could be collected (Fig. 6, scan No. 3), yielding a good average composition and the result that it has no perceptible homogeneity region, the EPMA determination limit of which is estimated to be ca. 0.4 at% N by using the formula [13]

$$\Delta c \approx \frac{2.33 c\sigma}{\sqrt{nN}} \qquad (2)$$

for 95% confidence, where c is the composition (50 at% N), n is the number of measurement points, N is the net number of accumulated counts for one measurement point and σ is the standard deviation ($\sigma = \sqrt{N}$). For $nN = 105000$ (10 data points with a net count rate of 1050 counts/s and 10 s counting time; Table 3, last line) $\Delta c = 0.36$ at% N.

Also the δ–TaN_{1-x} phase was accessible to a measurement of the homogeneity region by use of such specimen geometry (compare scan No. 1 with No. 2). This phenomenon was especially exploited for the characterization of phases in the Hf-N system [4] and the ζ phases within the group V transition metal-carbon systems. These ζ phases form very thin diffusion bands, which in some cases can even escape observation in the light-optical microscope in semi-infinite samples, but have a greatly increased width when the sample thickness decreases. The EPMA scans where the ζ phases are included are shown in Fig. 7a–c. For the ζ-Nb_4C_{3-x} phase the homogeneity range is 40.1 – 40.7 at % C and for the ζ-Ta_4C_{3-x} phase 38.2 – 39.0 at % C, whereas for ζ-V_4C_{3-x} the homogeneity range is practically zero and a composition of 38.0 at% C was measured (these data were collected from a variety of line scans). We believe that the results of the present technique are to be preferred over data from bulk chemical analyses of samples that did not consist of a single phase (the very existence of ζ-Nb_4C_{3-x} was even in doubt [14]).

Conclusion

Multiphase diffusion couples are well suited for studying both the homogeneity ranges of phases and the diffusion kinetics, provided that an accurate chemical analysis of the diffusing species is possible. Carbides and nitrides of the heavier transition metals, e.g. TaC or HfN, pose some problems, since EPMA utilizes low-intensity soft X-ray radiation in conjunction with small weight percentages of light elements.

Relief effects give rise to difficulties near the interfaces between different phases and at the edges of the specimens. The surface relief depends on the polishing procedure; lapping of the specimens largely overcomes this problem. The analytical total, i.e. the non-normalized sum of the concentrations of all independently determined constituents, provides a key to detect the presence of such relief effects, provided that quantification procedures are adequate.

For the analysis of phases which generally develop phase bands with a width of only a few μm and which in general cannot be analysed by EPMA for their homogeneity range it could be shown that the analytical determination of even small homogeneity ranges is possible using a restricted sample geometry for the preparation of diffusion couples, which enhances the widths of diffusion bands.

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