

X-ray mapping of microstructures in hardmetals and cermets

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INTRODUCTION

Hardmetals and cermets are alloys with hard phase constituents such as WC, TiC, Mo₂C, TiN, ... and its combinations, together with a binder phase. Phase reactions and diffusion occur upon sintering of such materials yielding a dense microstructure. In the present work the behaviour of different hard phases was investigated by means of solid/solid diffusion couples of the type Ti(C,N)/(Ti,W)C and Ti(C,N)/(Ti,Mo)C in order to study the diffusion processes in the hard phases of such materials. From sintering experiments [1] the core-rim structure of the hard phase particles was attributed to the presence of a miscibility gap in both systems (Fig. 1).

EXPERIMENTAL

Sample preparation: Onto hot-pressed Ti(C,N) different mixed hard-phase powders containing Mo and W were hot-pressed in Ar at 2200°C and ca. 620 bar. The composition of the couples was Ti(C_{0.7}N_{0.3})/(Ti_{0.7}W_{0.3})C, Ti(C_{0.8}N_{0.2})/(Ti_{0.8}Mo_{0.2})C and Ti(C_{0.7}N_{0.3})/(Ti_{0.7}Mo_{0.3})C. These samples were annealed at 1 bar Ar above ambient pressure at 1500°C for 1008h and 1512h respectively. The samples were cut with a diamond disk, embedded in cold-setting resin and ground with a 20µm diamond disk. After this procedure a 3µm diamond paste was applied on a hard cloth for polishing.

EXPERIMENTAL

Microprobe investigations: The microprobe investigations were performed with a CAMECA SX 50 microprobe. Chemically analysed carbide and nitride standards were

used [2]. A variety of different conditions was chosen for the beam current, voltage, type of crystal and background position to carry out the measurements (see Tab.1). To avoid the problem of peak coincidences and reduce the problem of contamination during measurements, N was subtracted from Ti as well as C from all elements. For X-ray mapping the scanning time per point and per element was 50ms. It should be noted that the X-ray mapping was not quantified, meaning that the same colour in different images is not equal to the same concentration.

Tab.1
EPMA conditions for the systems (Ti,W)(C,N) and (Ti,Mo)(C,N)

Element*	Beam conditions [kV/nA]	Crystal*	Peak Position [10 ³ sin ² θ]	+bg* [10 ³ sin ² θ]	-bg* [10 ³ sin ² θ]
W	10/100	TAP	27176	820	530
C	10/100	PC1	74125	7000	7000
Ti	10/100	PET	31424	800	600
Mo	10/100	PET	61787	600	1.2
N	10/100	PC1	52998	5000	4500

* PC1: 59.6 A, PET: 8.742A, TAP: 25.9A

* Background to be added to (+bg) or subtracted from (-bg) the peak position

* All the measurements were carried out with cold trap and without oxygen jet

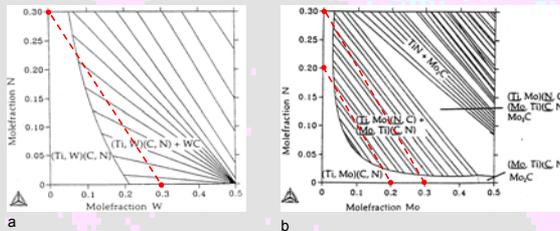


Fig.1
Isothermal sections at 1450°C of the systems Ti-W-C-N (a) and Ti-Mo-C-N (b); the red dots mark the starting compositions of the diffusion couples;

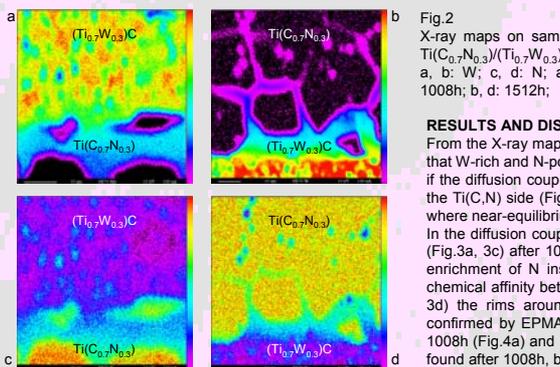


Fig.2
X-ray maps on sample Ti(C_{0.7}N_{0.3})/(Ti_{0.7}W_{0.3})C; a, b: W; c, d: N; a, c: 1008h; b, d: 1512h;

Fig.3
X-ray maps on sample Ti(C_{0.8}N_{0.2})/(Ti_{0.8}Mo_{0.2})C; a, b: Mo; c, d: N; a, c: 1008h; b, d: 1512h;

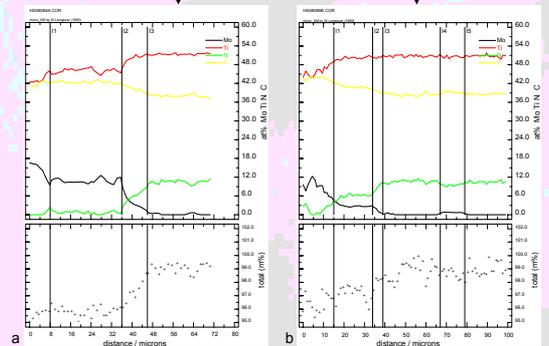
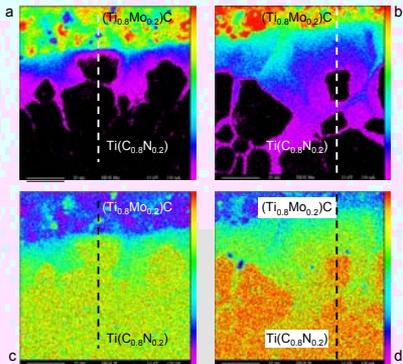


Fig.4
EPMA line scans across sample Ti(C_{0.8}N_{0.2})/(Ti_{0.8}Mo_{0.2})C a: over a distance of 80µm annealed for 1008h; b: over a distance of 100µm annealed for 1512h; 11-15: concentration changes according to the grains identified in Fig. 3;

RESULTS AND DISCUSSION

From the X-ray maps made at the interface of the diffusion couple Ti(C_{0.7}N_{0.3})/(Ti_{0.7}W_{0.3})C it can be observed that W-rich and N-poor rims were built after 1008h (Fig.2a, 2c). The thickness of these rims remains constant if the diffusion couple is annealed for further 504h, while W diffuses deeper along the grain boundaries into the Ti(C,N) side (Fig.2b, 2d). Evidently, the core-rim structure remains stable at positions near the interface where near-equilibrium conditions are reached.

In the diffusion couple Ti(C_{0.8}N_{0.2})/(Ti_{0.8}Mo_{0.2})C a core-rim structure was built by diffusion of Mo into Ti(C,N) (Fig.3a, 3c) after 1008h. Hence Mo-rich rims were built around the Ti- and N-rich zones (Fig.2a). Again an enrichment of N inside the Mo-poor parts is clearly visible (see Fig.3c and 3d). This is due to the low chemical affinity between Mo and nitrogen. Upon annealing the diffusion couple for additional 504h (Fig.3b, 3d) the rims around the Ti- and N-richer parts grew and thus became thicker. This observation was confirmed by EPMA line scans made across the diffusion zone of the diffusion couple after annealing for 1008h (Fig.4a) and 1512h (Fig.4b). In the second diffusion couple (see Fig.1b) also a core-rim structure was found after 1008h, but the thickness of these rims did not change upon annealing time.

CONCLUSION

We conclude that in the Ti-W-C-N system a stable core-rim structure is formed because of the miscibility gap. In the Ti-Mo-C-N system this miscibility gap (Fig.1b) was not found to be stable at the composition Ti(C_{0.8}N_{0.2})/(Ti_{0.8}Mo_{0.2})C and thus the sample homogenises to a single phase near the interface (as opposed to a thermodynamic modelling study [1]).

REFERENCES

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