

SiC bonded Diamond composites produced by reactive infiltration with silicides

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Aim

Pressure less preparation of superhard wear resistant diamond material by reactive infiltration with silicides:

- possibility of complete infiltration with silicides
- possibility of reduced residual Si content in the final material in comparison with Si infiltration
- special feature of microstructure formation

Background

Table 1: Overview of eutectic and melting compositions in Si-transition-metal systems

Phase diagramm	Eutectic Phases	T _{eutectic} in °C	Eutectic Comp. Mol % metal	Max metal content in the liquid at 1600°C in Mol%	Source (ASM international)
Si-Ti	Si-TiSi ₂	1300	16.0	40	90766
Si-Zr	Si-ZrSi ₂	1346	8.5	20	107146
Si-Hf	Si-HfSi ₂	1329	11.0	25	105058
Si-V	Si-VSi ₂	1400	3.5	20	90211
Si-Ta	Si-TaSi ₂	1400	1.5	7	981062
Si-Mo	Si-MoSi ₂	1406	3.0	5	107122
Si-Cr	Si-CrSi ₂	1328	15.0	53	1600374

Ti and Cr have the largest range of compositions suitable for infiltration. Evaluation of the infiltration and phase formation with Ti-, V – silicides.

Preparation and analysis

Preparation

- Mixing diamond 50 μm/10 μm with volume ratio 70/30 with phenolic resin in ethanol, drying in the rotavap,
- Uniaxial pressing of tablets Ø 30 mm height appr. 5 mm
- Infiltration in vacuum at 1525- 1600 °C (heating rate 50 K/min, isothermal dwell time 15 min) with eutectic mixture TiSi₂/ Si / VSi₂Si

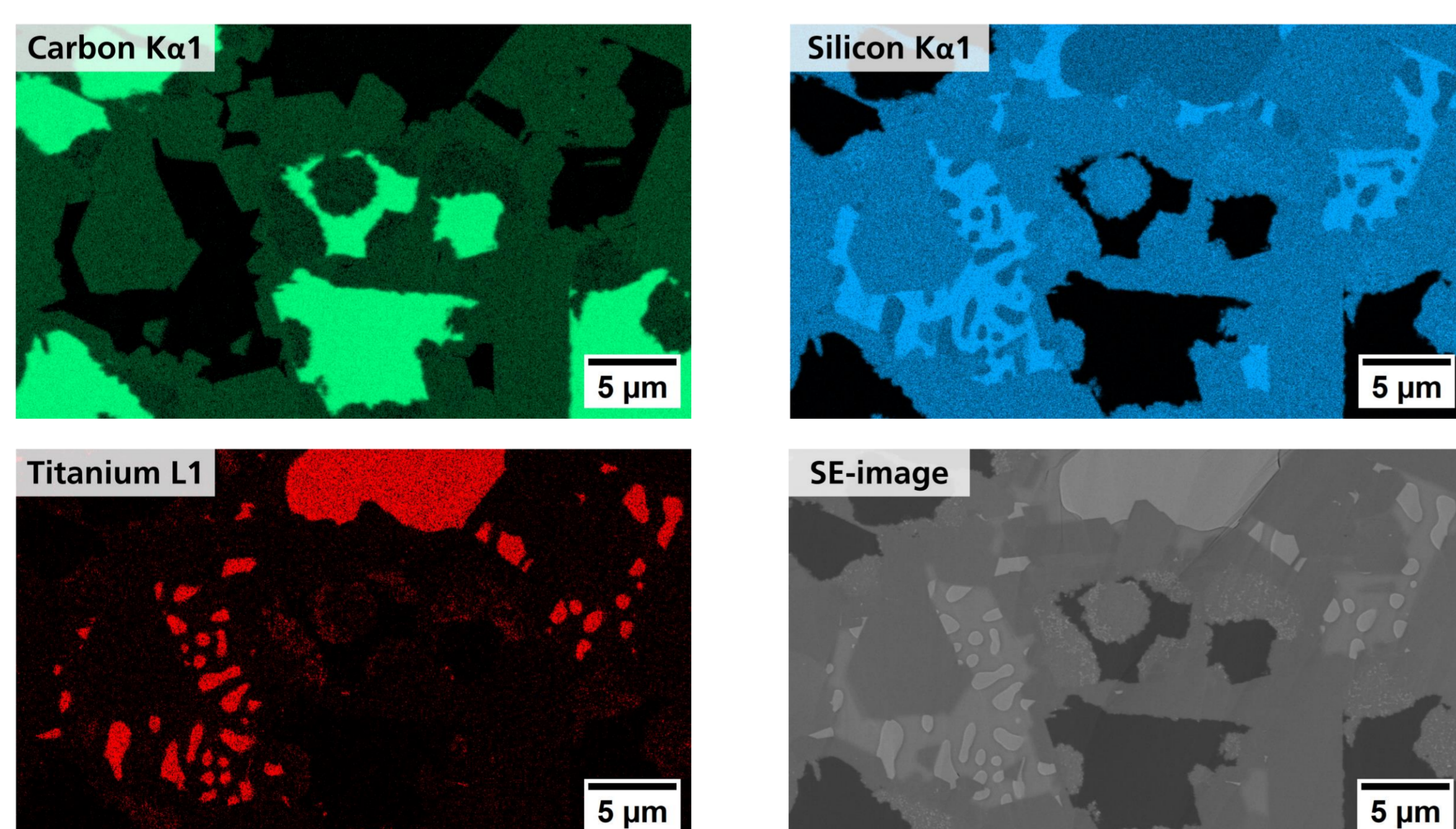


Fig. 1 : Results of the EDX mapping of the infiltrated with the eutectic composition Si/TiSi₂ at 1525°C material (eutectically solidified melt is clearly visible next to μm size silicide precipitates)

Analysis

- Density (DIN EN ISO 18754:2022-06)
- X- ray phase analysis (D8 Advance CuKα- radiation)
- Microstructure preparation: ion beam polishing (BalTec RES 101)
- Microstructure: FESEM Crossbeam 550 with EDX Ultim® Max 170 and EBSD detector (Symmetry S2)

Table 2: Density and phase content after infiltration*

Sample Name	Infiltration		Density in g/cm ³	Phases	
	Metal	Temp. in °C		Surface	Volumen
Si/Dia	Si	1525	3.27	Si	Si
TiSi ₂ /Si/Dia-1	TiSi ₂ / Si	1525	3.42	TiSi ₂ / Si	Ti ₃ SiC ₂ /TiSi ₂
TiSi ₂ /Si/Dia-2	TiSi ₂ /Si	1575	3.44	TiSi ₂ /Si/ T ₃ SiC ₂	Ti ₃ SiC ₂ / TiSi ₂ /TiC
TiSi ₂ /Dia	TiSi ₂	1575	3.57	Ti ₃ SiC ₂ / TiC	TiC/ T ₃ SiC ₂
VSi ₂ /Si/Dia	VSi ₂ /Si	1525	3.34	Si/VSi ₂	Si/VSi ₂

*(phases beside the main phase SiC and diamond)

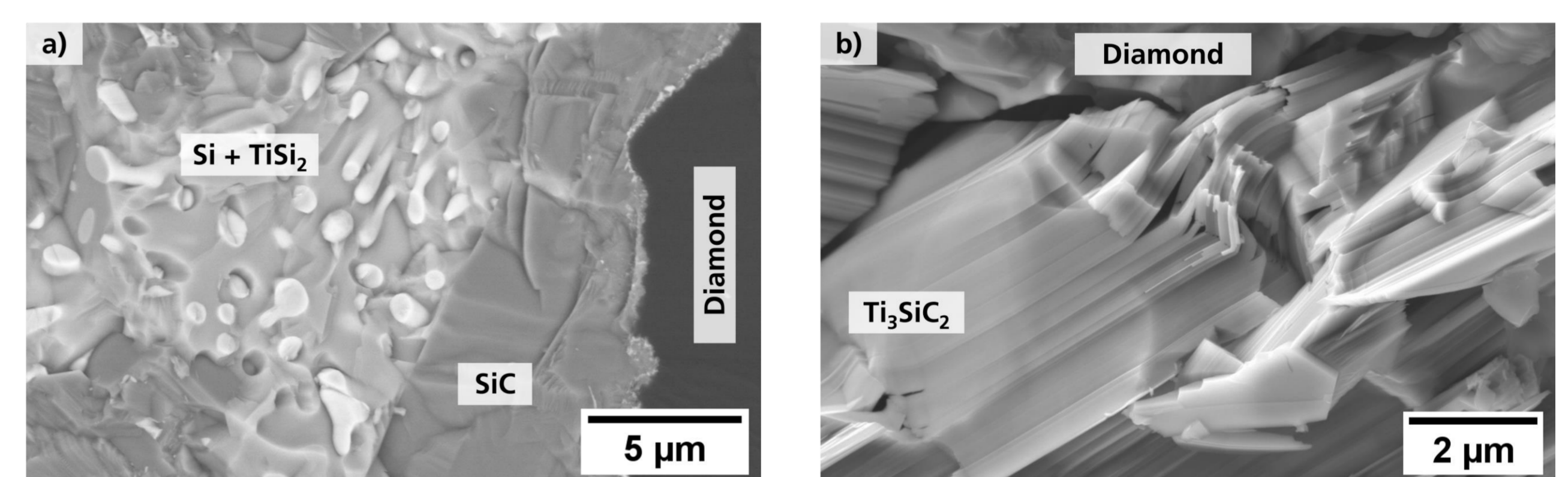


Fig. 2: FESEM images of the fracture surface a) surface b) volume (In the near surface area clearly the TiSi₂ inclusions are visible , in the volume the kinking of the MAX-phase is observed)

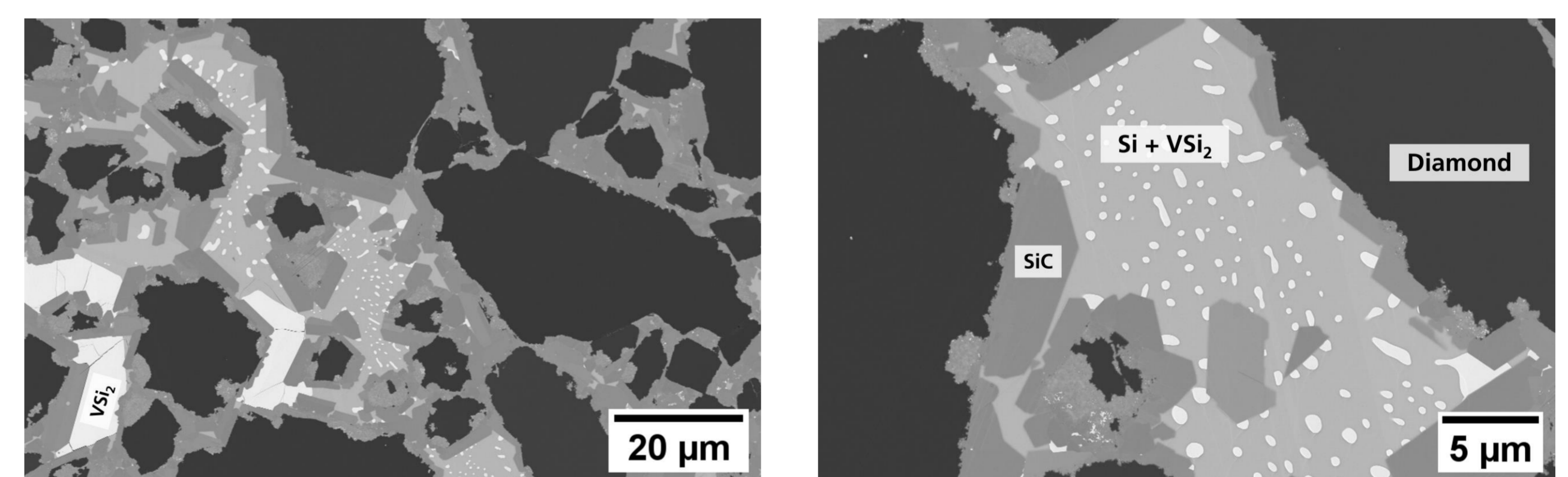


Fig. 3 : FESEM images of the fracture surface of sample VSi₂/Si/Dia in the near surface area clearly the TVSi₂ inclusions are visible , around large VSi₂ crystals local cracks are formed

Results

- Full infiltration is possible (Tab. 2)
- Formation of Ti₃SiC₂ (MAX-Phase) ; TiSi₂, TiC,
- dependent on the composition of the melt gradients are formed (Tab. 2 ; Fig. 1-2)
- residual Si content is strongly reduced in comparison with Si infiltration
- The fracture surface reveal possible additional strengthening effects (Fig. 2)
- During infiltration with VSi₂/Si no carbide and MAX phase formation was observed; beside eutectic structures large VSi₂ crystals are formed around large MSi₂ crystals microcracks are formed (Fig. 3)
- Further investigation of the reactions during infiltration and gradient formation and of the properties of the resulting materials is necessary.