

TEM Analysis of Diffusion-Bonded Silicon Carbide Ceramics Joined Using Metallic Interlayers

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1. Introduction

properties and applications of SiC (SA-THX)

purpose of diffusion bonding

2. Sample preparations used for diffusion bonding

Substrates : SA-TyrannohexTM (SA-THX)

Interlayers : Ti-Mo, Ti-Cu foil

3. Experimental results

STEM images of the bonding area

TEM images and SAED patterns of the reaction compound

4. Discussion about the microstructure of the formed phases by diffusion bonding

5. Summary

SiC fiber-bonded ceramics, SA-Tyrannohex[®]

SiC composite material

1. Excellent mechanical properties
2. Good oxidation resistance
3. High thermal stability

Especially,

SA-Tyrannohex (SA-THX)

... SiC fiber-bonded ceramics

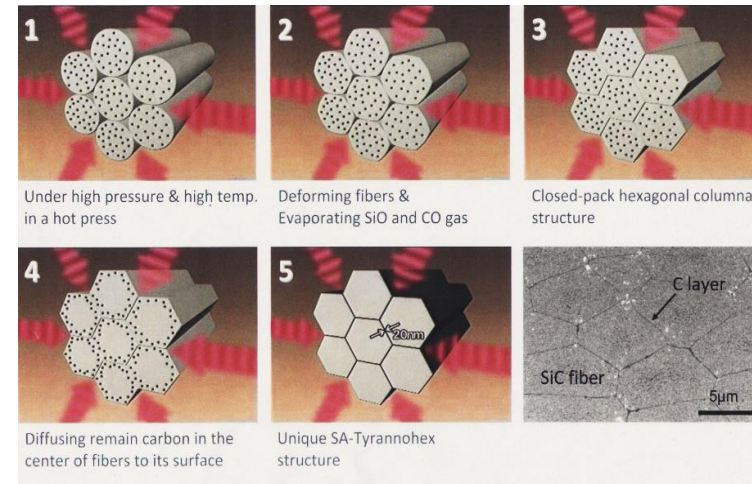
- High strength sustained **up to 1600°C in air**
- **High fracture toughness (1200 J·m⁻²)**

⇒ Promising material for high-temperature and extreme environment applications

e.g. injector applications, combustion liner, nuclear fusion reactor and turbine engine applications



http://www.ube-ind.co.jp/japanese/products/chemical/chemical_19.htm



T. Ishikawa et al, *Science*, 282, 1295-1297 (1998).
T. Ishikawa et al, *Nature*, 391, 773-775 (1998).

For wide range uses of SA-THX

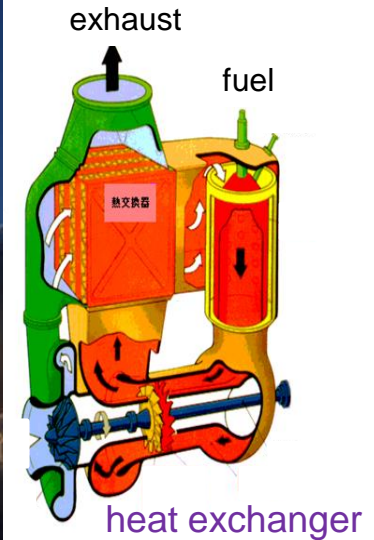
However, geometrical limitations hinder the wide use of SA-THX. It is difficult to fabricate large, or complex shaped components by Hot Pressing or CVD.

Therefore, new advanced methods are needed.

Under those circumstances,

One cost-effective solution for fabricating large, complex-shaped components is the **joining** of simple shaped ceramics.

In this study, we are going to focus on diffusion bonding.



Diffusion Bonding of SA-THX using metallic Interlayers

Used sample

@NASA

substrate: SA-THX ...SiC fiber-bonded ceramics, UBE Industries

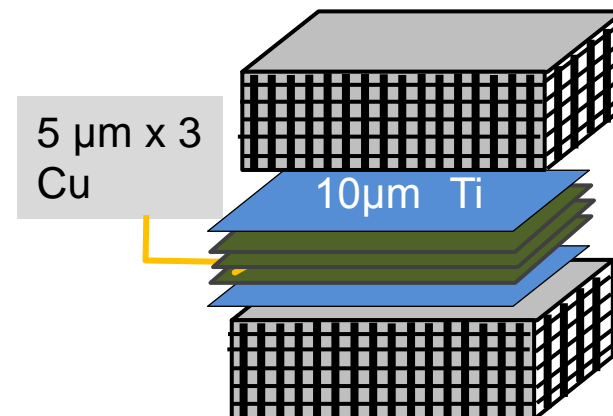
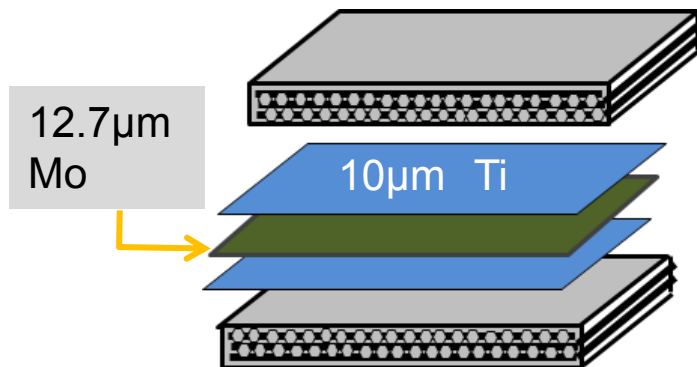
metallic interlayer: Ti-foil, Mo-foil and Cu-foil, Goodfellow Corporation

Bonding process

Hot-press in 1200°C, 4hour, vacuum 30MPa

Ti-Mo foil

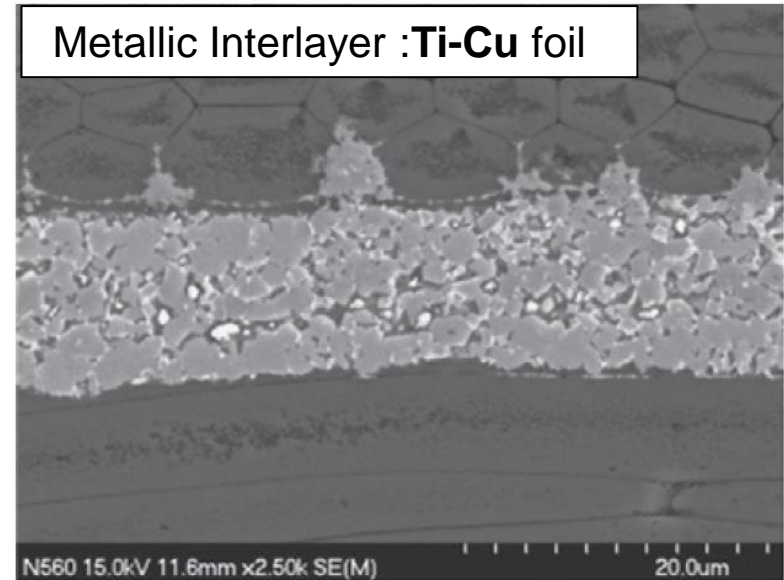
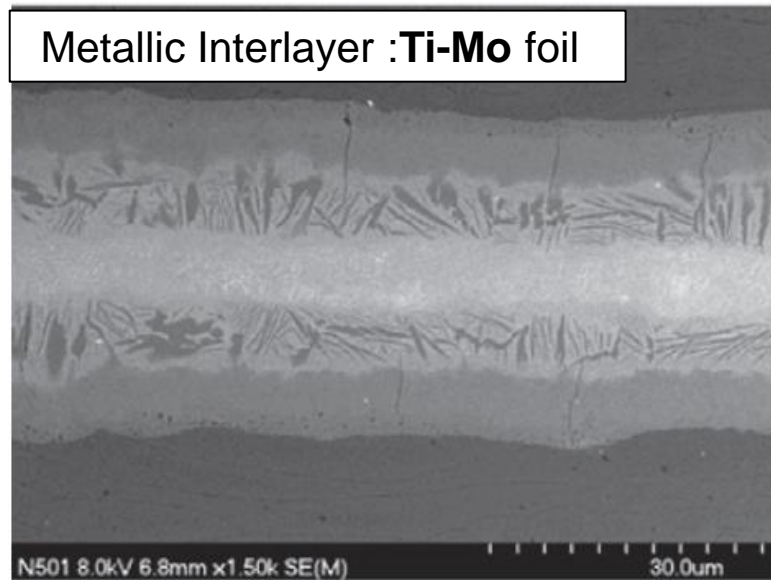
Ti-Cu foil



10µm Ti and 12.7 µm Mo interlayer

10µm Ti and 15µm Cu interlayer

Diffusion Bonding of a SA-THX using metallic Interlayers



Knoop hardness of diffusion bonded joints.

	Average HK (\parallel joint)	Average HK (\perp joint)
SA-THX/Ti/Mo/Ti/SA-THX	<u>717.7 ± 273.6</u>	758.9 ± 299.3
SA-THX/Ti/Cu/Ti/SA-THX	<u>816.5 ± 43.9</u>	—
SA-THX (un-bonded)	1244 ± 176	624 ± 205

M.C. Halbig, et. al., Ceramics International 41(2015)2140–2149

Objectives

We diffusion bonded SiC and SiC (SA-THX and SA-THX) using Ti/Cu foil metallic interlayer.

We carried out TEM and STEM observations with the diffusion bonded sample prepared by FIB technique.

1. Evaluate microstructures of the diffusion bonding area by TEM and STEM.
2. Characterize the reaction compound in the diffusion bonding area by STEM-EDS and SAED analysis.

Experiment (FIB and STEM)

Focused Ion Beam, FIB
(Hitachi FB-2200)



Cs-corrected STEM
(Hitachi HD-2700)



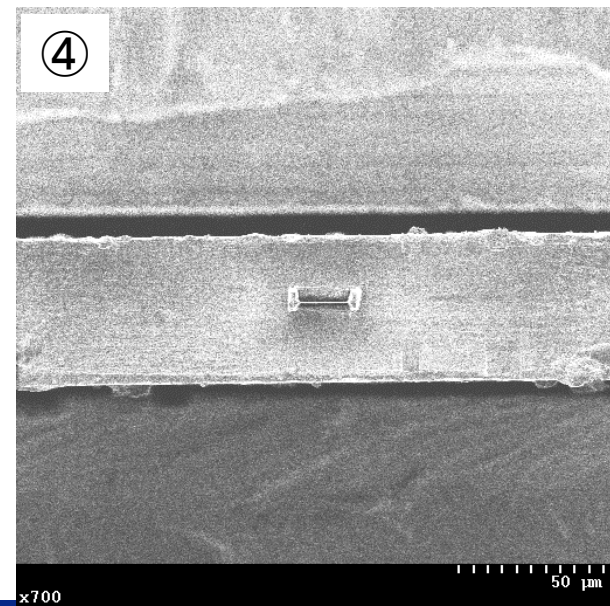
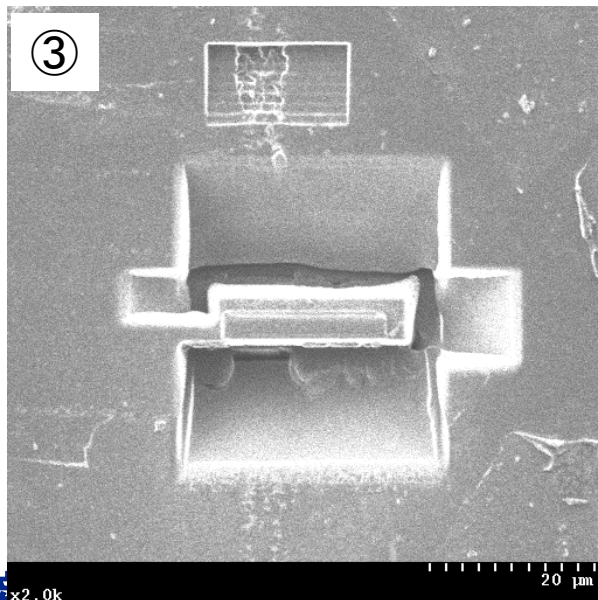
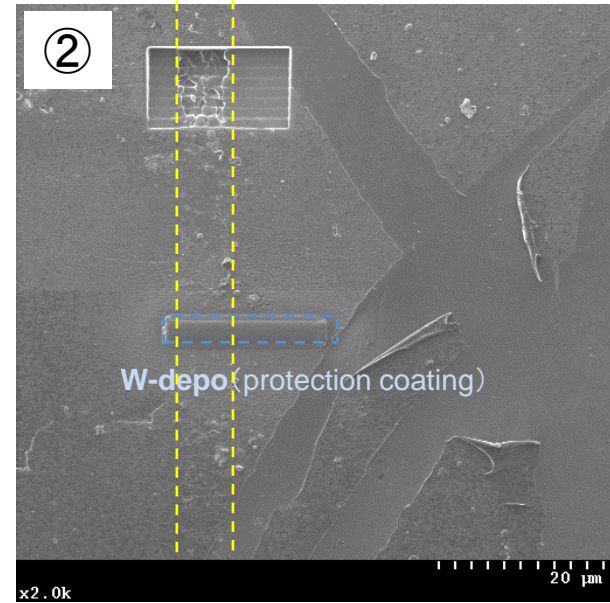
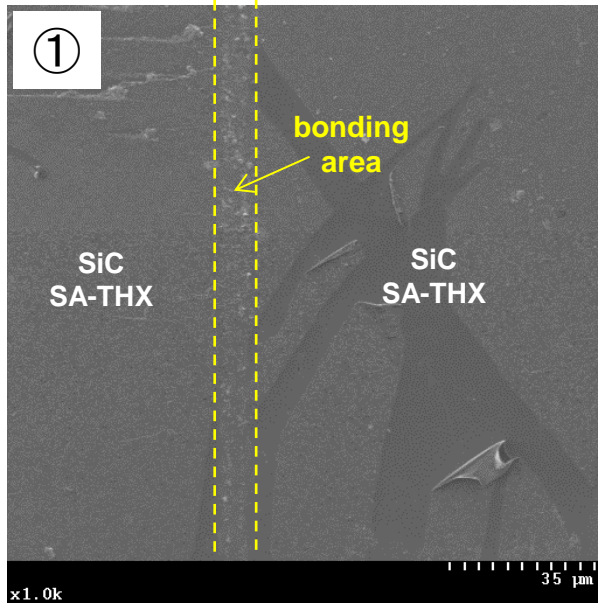
Prepared thin samples for TEM and STEM.

Checked the thin samples prepared by FIB.

Three-Observation mode:

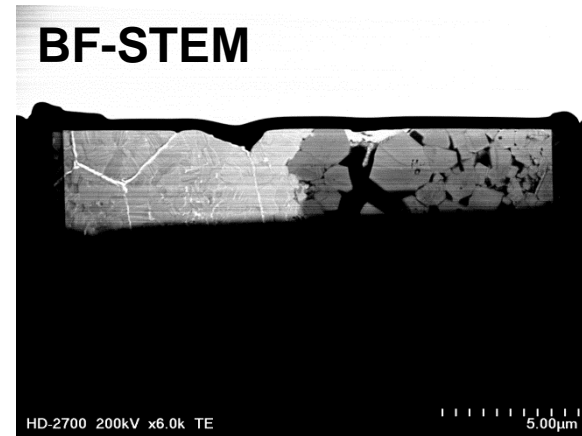
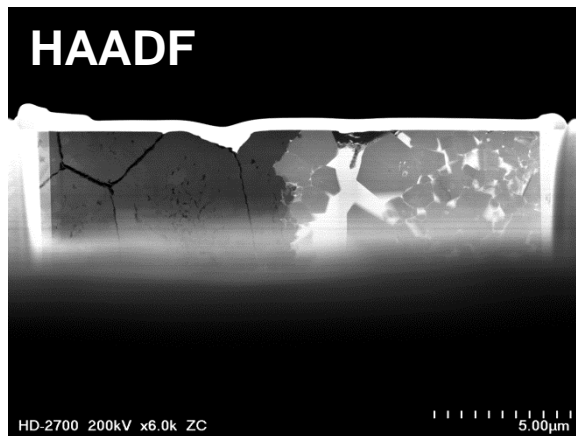
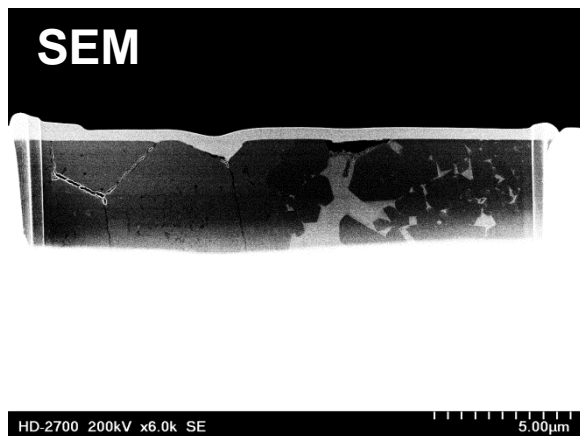
SEM, BF-STEM and HAADF

Fabricating procedure of the thin sample (SIM image obtained by FIB)

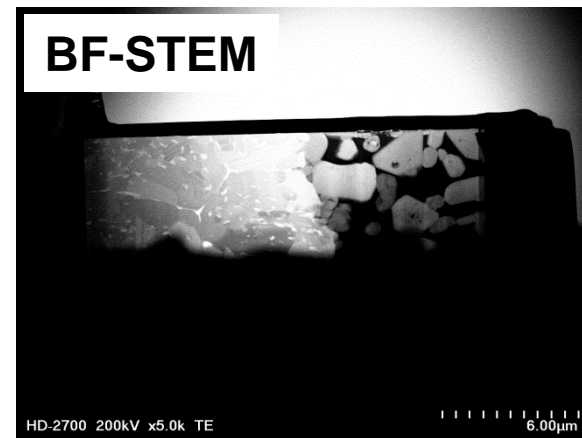
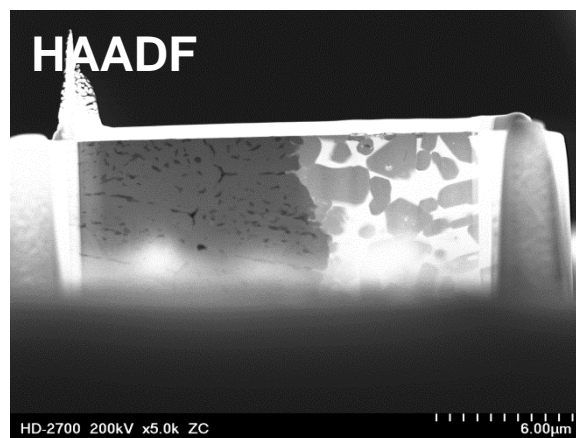
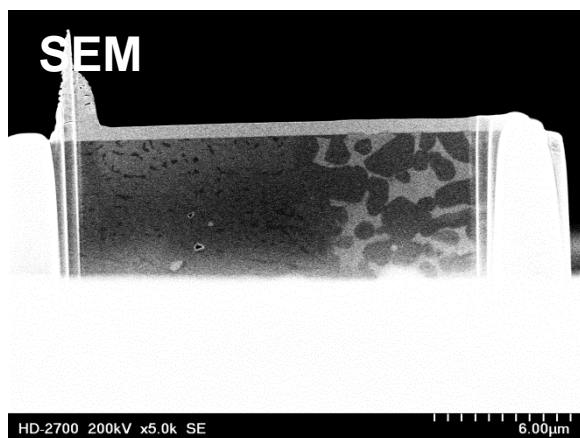


STEM observation of the FIB sample

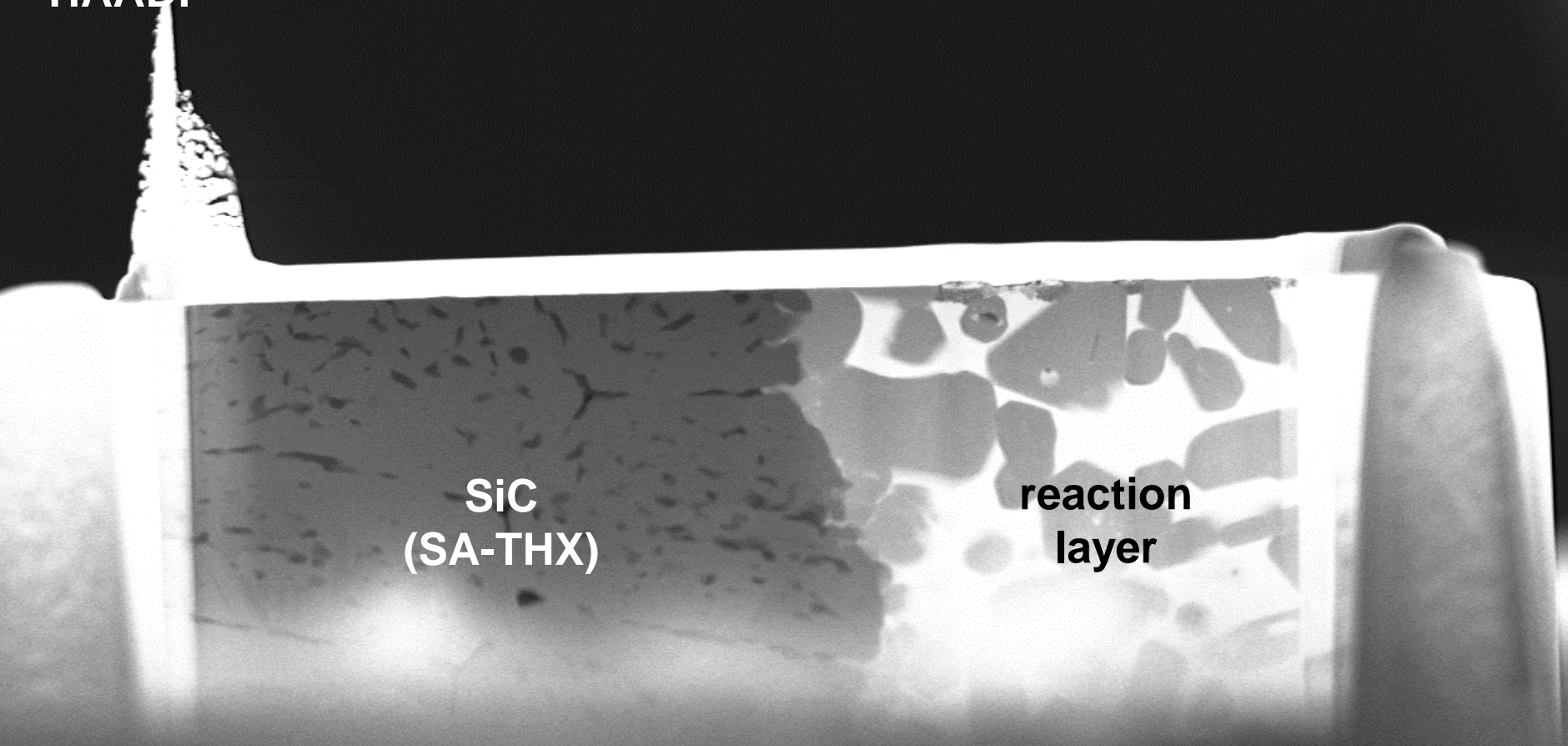
Ti/Cu interlayer **parallel** to SiC fiber



Ti/Cu interlayer **Perpendicular** to SiC fiber



HAADF



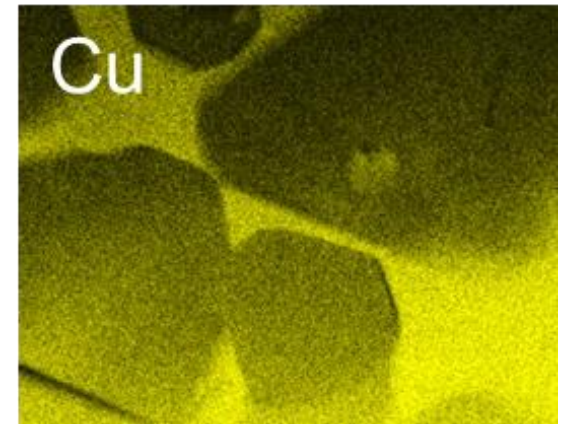
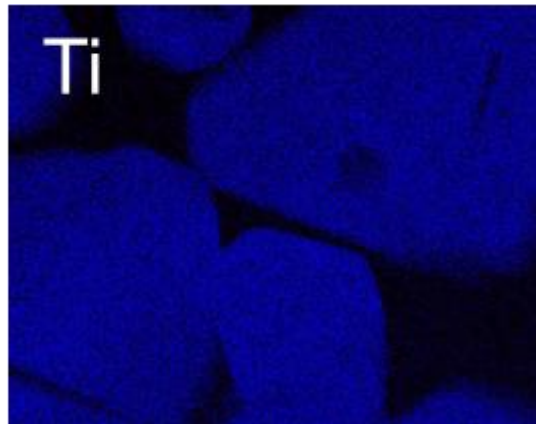
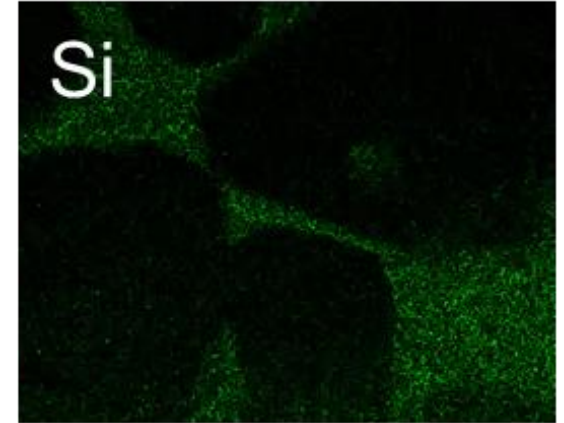
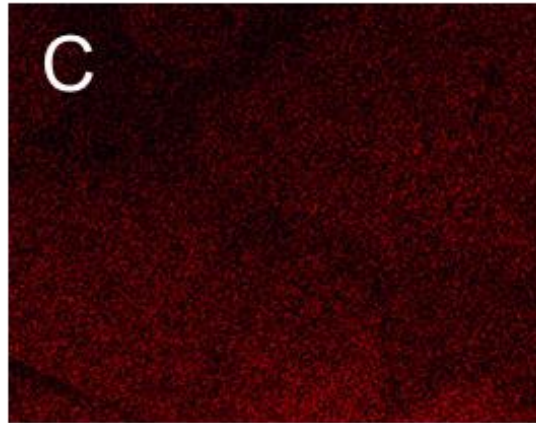
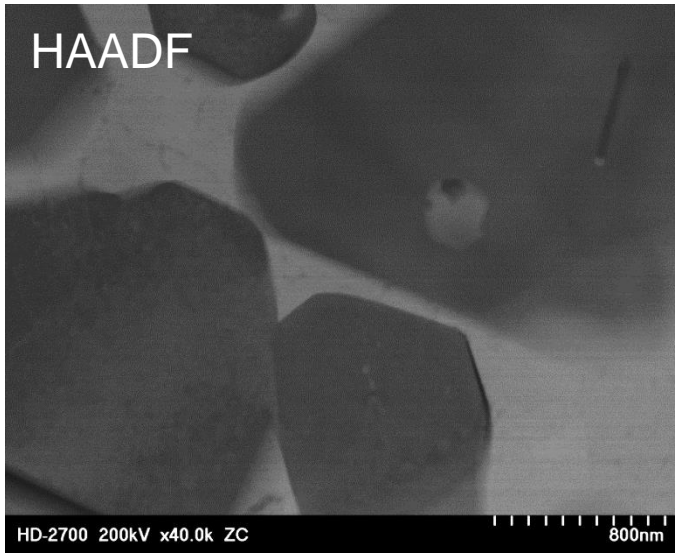
SiC
(SA-THX)

reaction
layer

- Only one layer in the bonding area
- No diffusion toward the side of SiC
- The reaction layer is composed of some grains

and matrix around the grains.

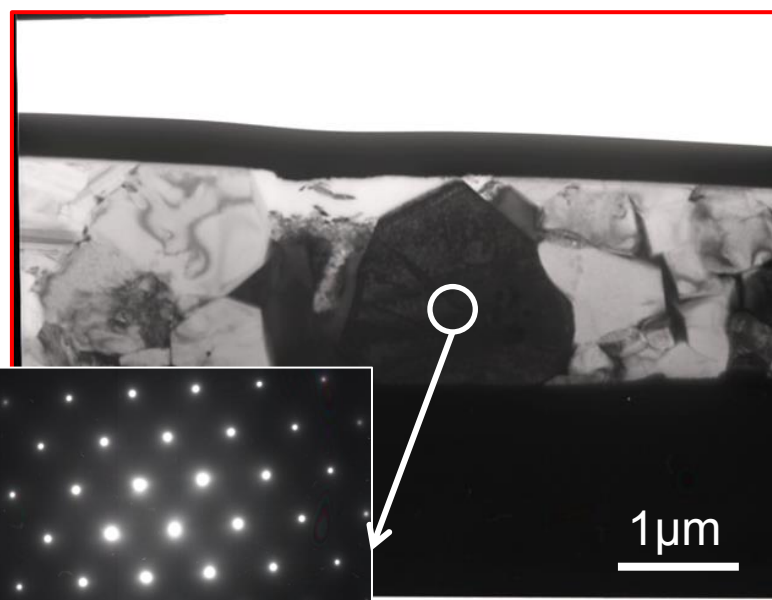
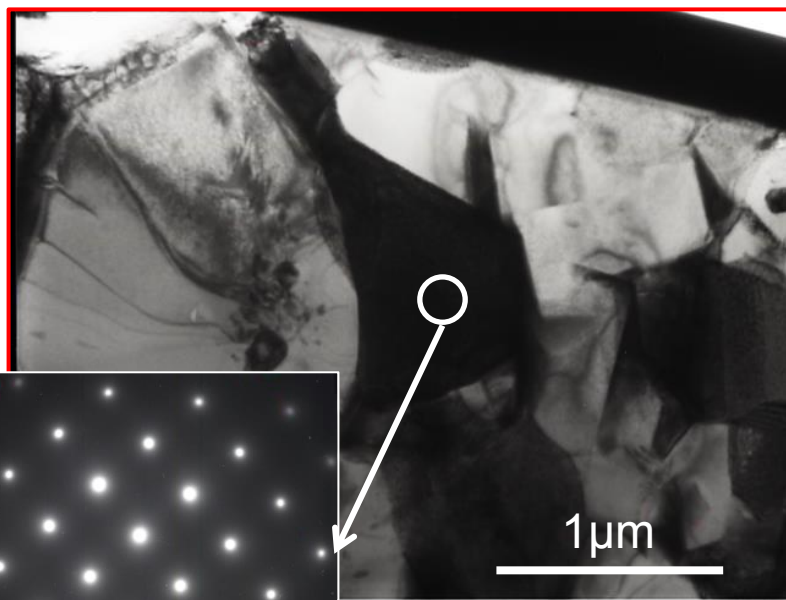
Element Mapping obtained by STEM-EDS analysis



* FIB mesh: Cu metal

Cu-Si matrix + precipitated TiC grains?

TEM image and SAD patterns of Ti-C compound

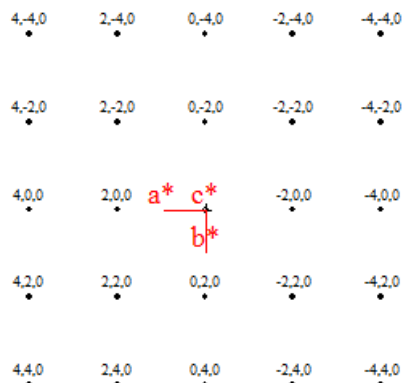


TiC

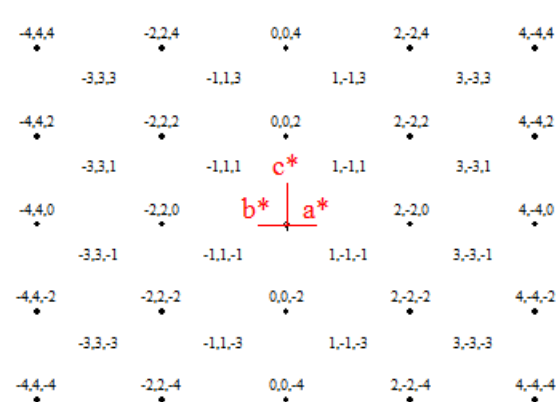
S.G.: Fm3m

NaCl-type

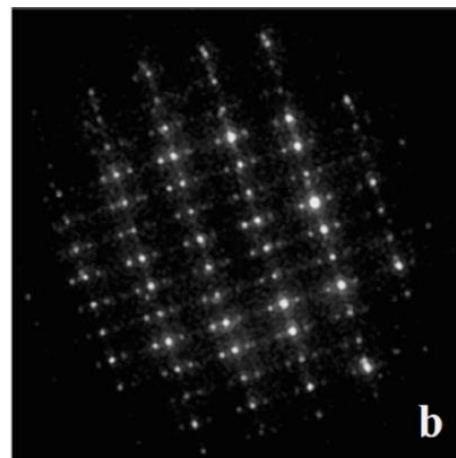
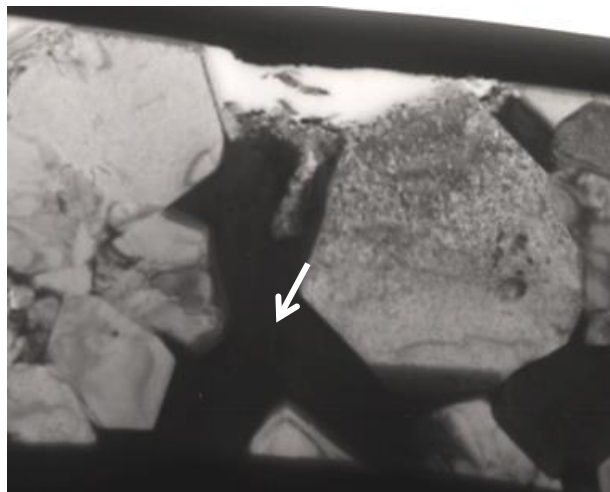
[001]TiC



[110]TiC



TEM image and SAD patterns of Cu-Si compound



SAD pattern of Cu_3Si
: η'' -phase
(RT phase of Cu_3Si)

//[001]

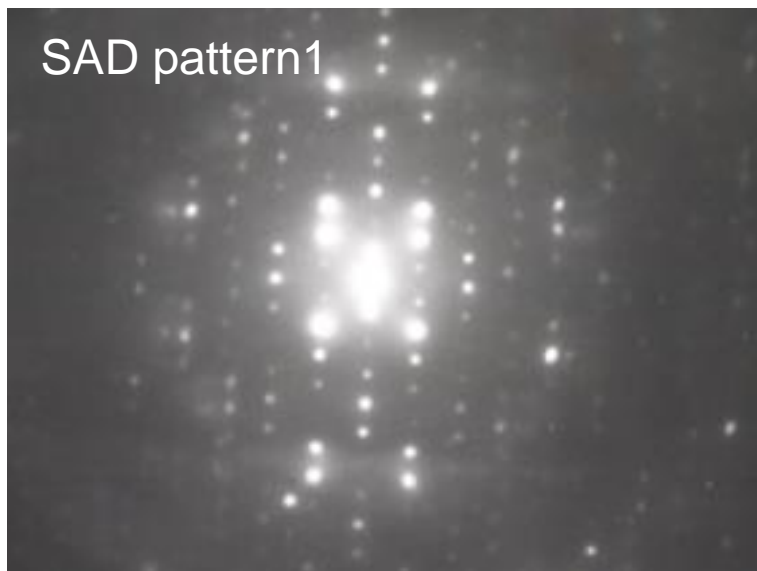
4 times superstructure

//[111]

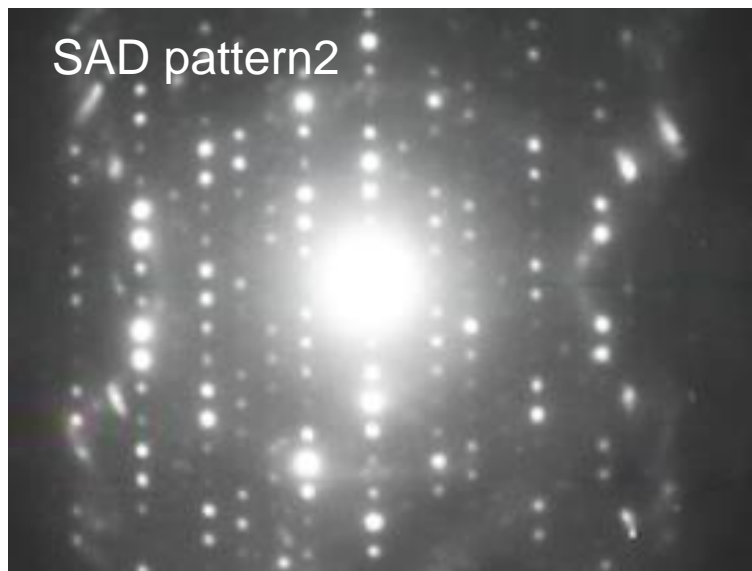
3 times superstructure

M. Heuer, *et. al.*, JAP 101, 123510 (2007)

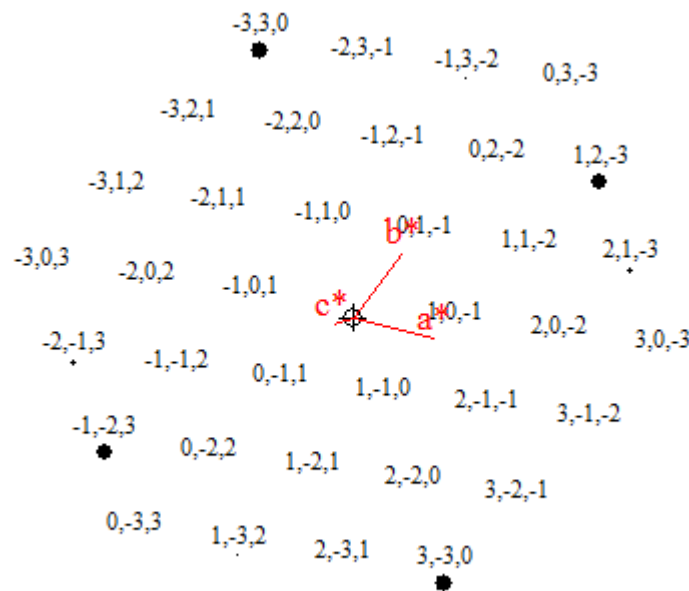
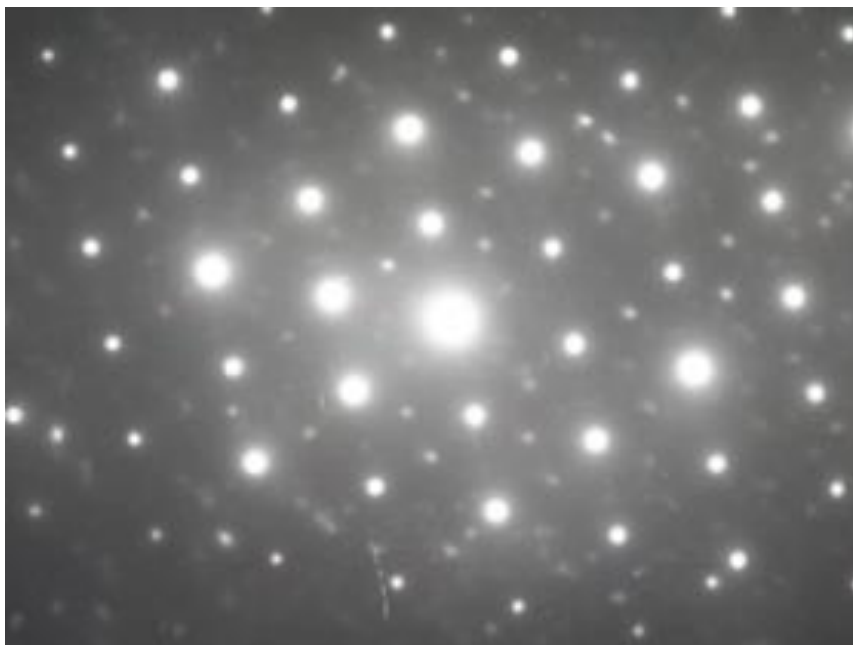
SAD pattern1



SAD pattern2



TEM image and SAD patterns of Cu-Si compound



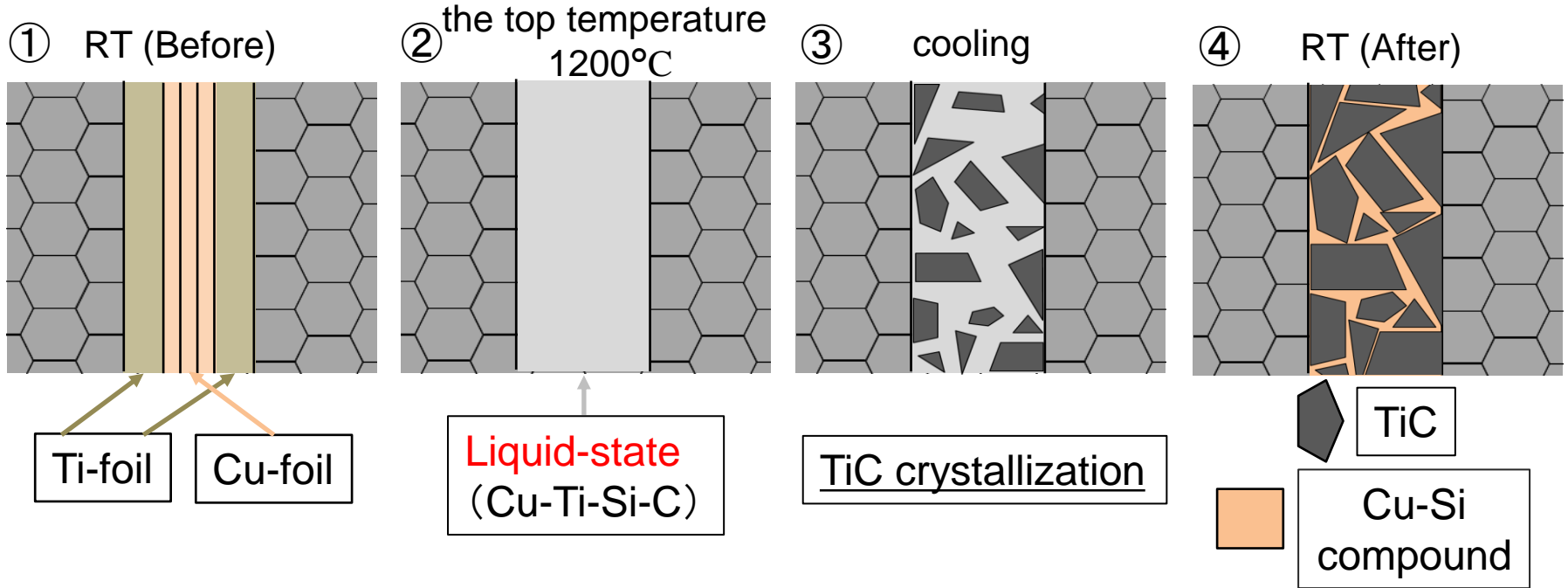
η -phase Cu_3Si :

(high temperature phase)

S.G.: P-3m1

$a=0.4091$ nm, $b=0.7358$

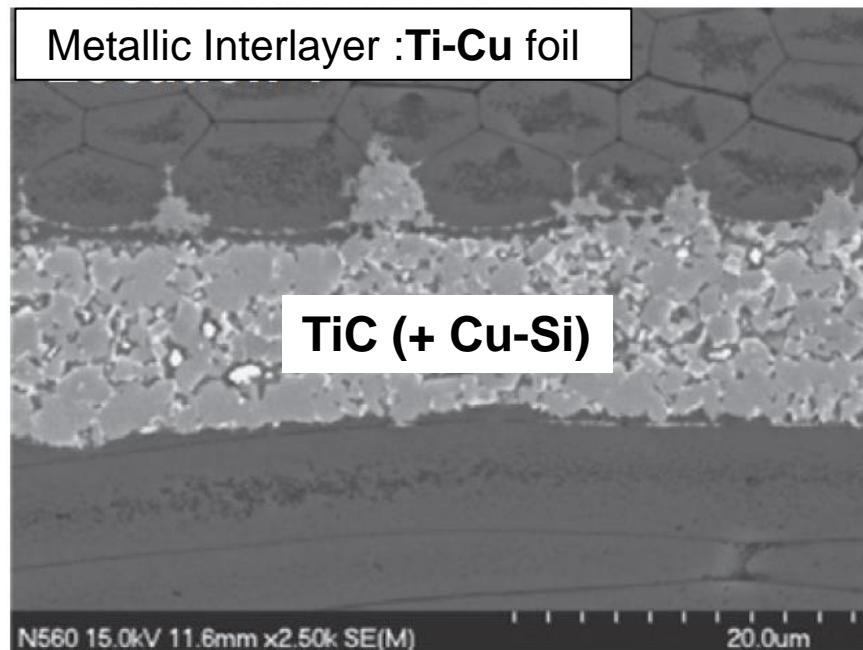
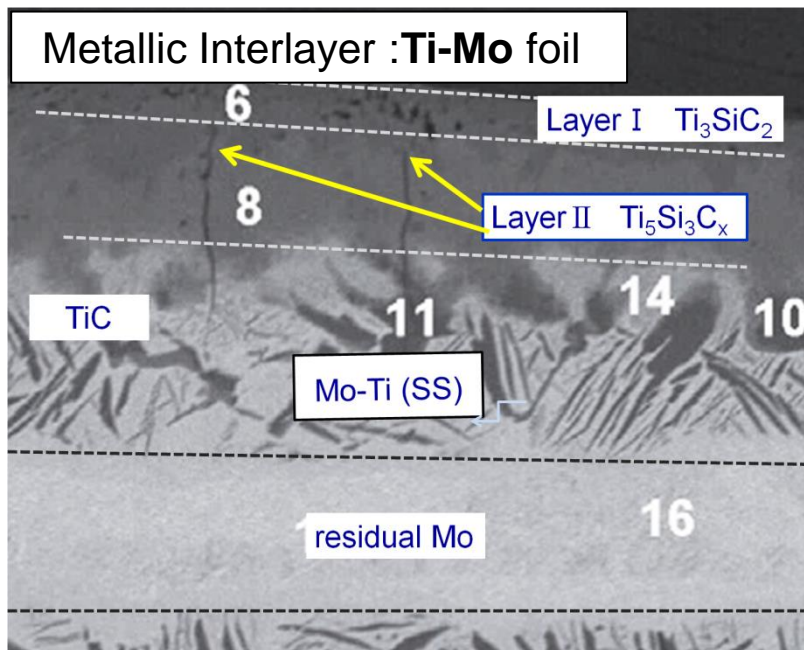
Consideration of the Results



The bonding layer is composed of TiC precipitations in Cu-Si matrix.

Cu-Si matrix plays a role of the binder of TiC precipitations?

Consideration of the Results (CTE)



coefficient of thermal expansion (CTE; α)

material	a-SiC	Mo	Ti	Cu	TiC	Ti ₃ Si ₅		Ti ₃ SiC ₂	
						a	c	a	c
CTE α ($10^{-6}K^{-1}$)	3.2	5.1	8.4	16.8	7.4	6.1	16.6	8.9	10

M.C. Halbig, et. al., Ceramics International 41(2015)2140–2149

Summary

- 1. We picked up thin samples from the bonded area of diffusion bonded SA-THX by a FIB micro-sampling technique. The prepared thin samples were sufficiently thin and less-damaged, and allowed the detailed evaluation by TEM and STEM.
- 2. The microstructure of diffusion bonded area was observed by STEM and TEM. The composition and crystal structures of the reaction compound were investigated by STEM-EDS and SAED method. The reaction layer of the diffusion bonding was composed of TiC precipitations in Cu-Si compound matrix.

This work was supported by JSPS KAKENHI
Grant Number JP16K06802.