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Effect of Ni-Nb Interlayer Thickness on Mechanical Property of HfB2 Composite Joints

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Background - Ultra High Temperature Ceramics

UHTCs：Ultra High Temperature Ceramics

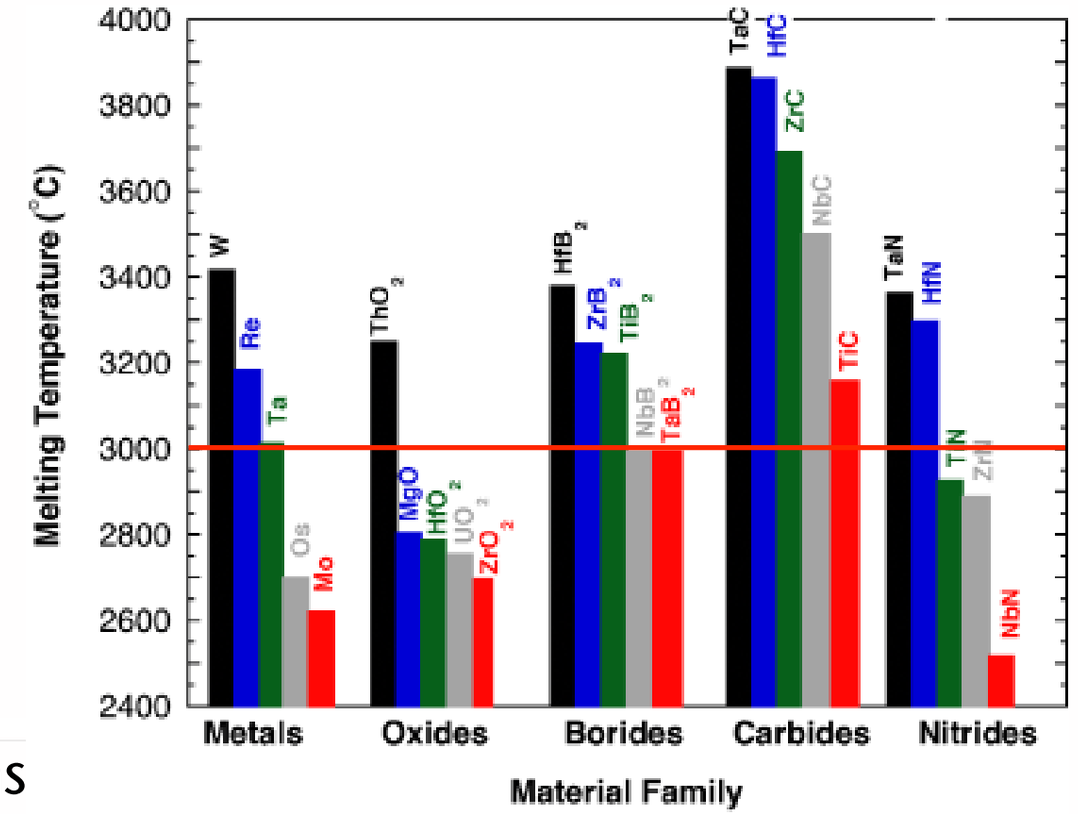
↳Several borides, carbides, and nitrides of the group IV and V metals

Ti, Zr, Hf, Ta **-B_2, -C, -N**

Excellent properties

- High melting points （> 3000℃）
- Good thermo-mechanical properties
- Chemical stability

→ Prospective applications



William G. Fahrenholtz et al. : J. Am. Ceram. Soc., 90 [5] 1347–1364 (2007)

Problems

- Poor sinterability and workability

→ It is difficult to produce the large and complex shaped parts of UHTCs.

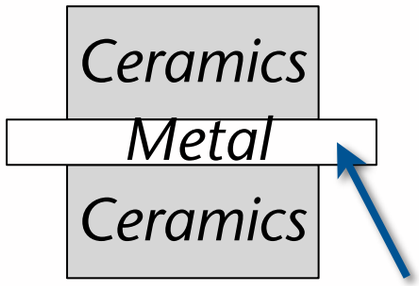
↓

For the practical use, an effective method of bonding will be required.

Background - Typical methods of ceramics bonding

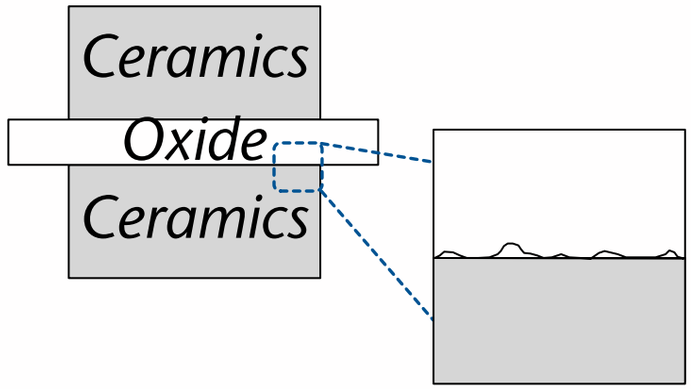
Typical examples of ceramics bonding

Metal brazing bonding



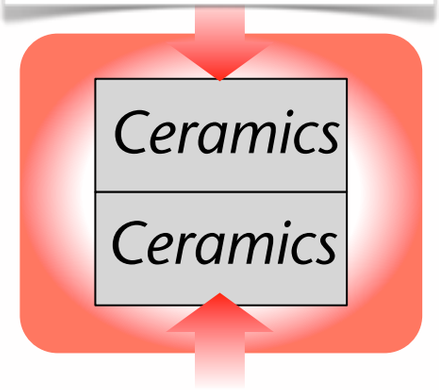
Low melting metal
↓
Heat resistance ×

Oxide solder bonding



Bad wettability
↓
Adhesion ×

Solid state bonding

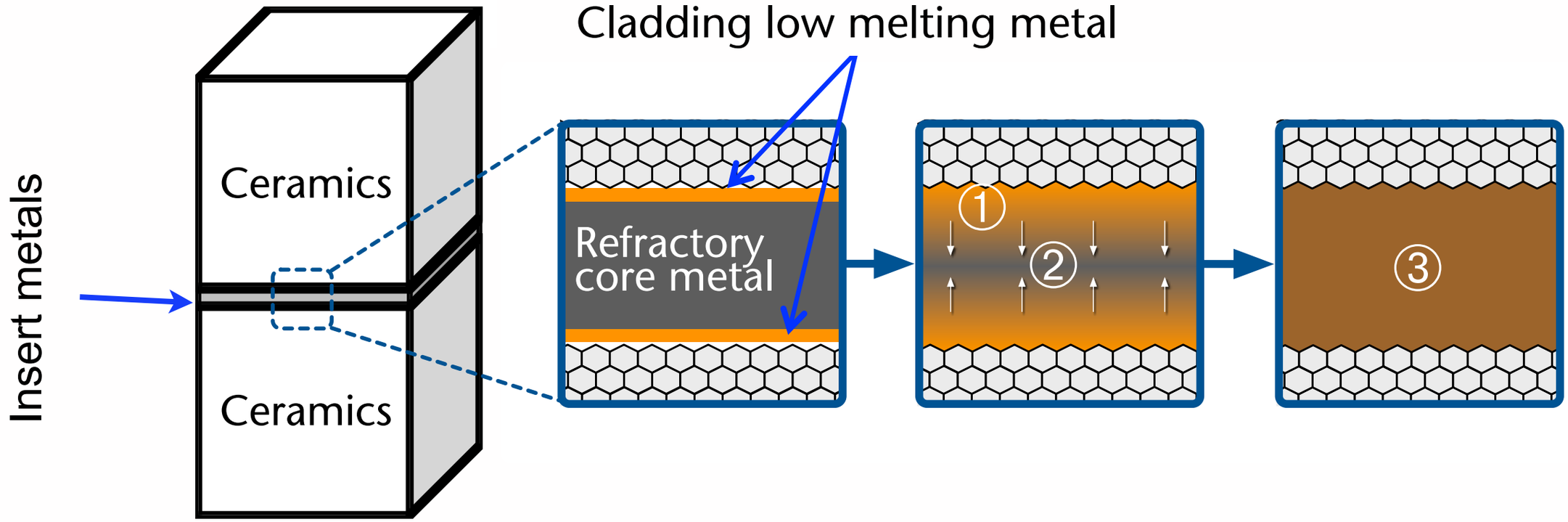


High temperature
and
high pressure
↓
Cost ×

Transient Liquid Phase (TLP) bonding

Background - TLP (Transient liquid phase) bonding

TLP bonding



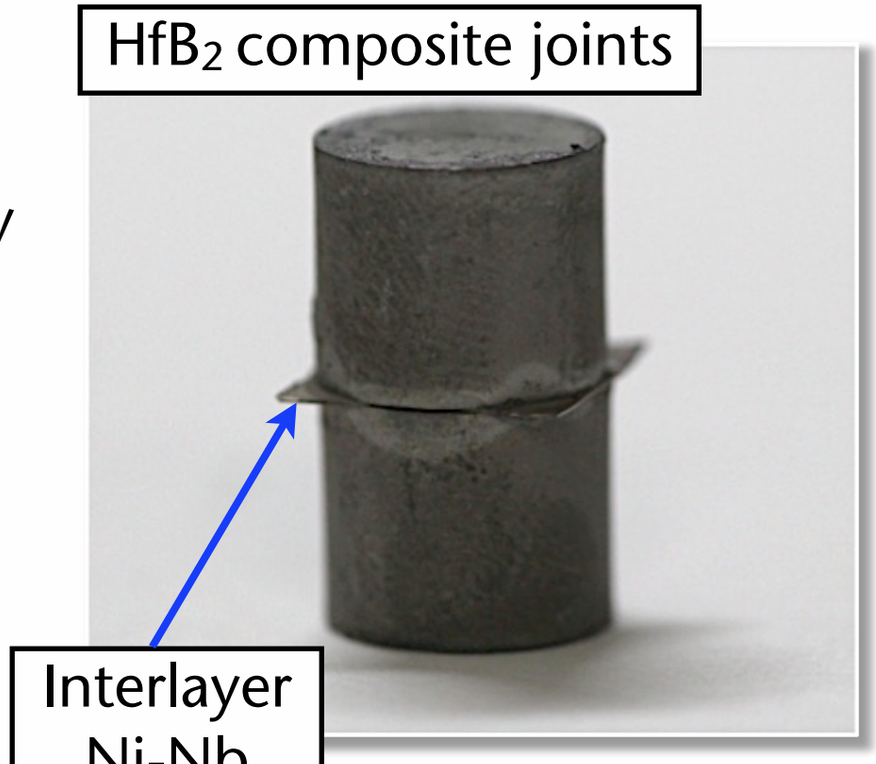
① The cladding metals will melt and fill the gap between the ceramics and the core metal.
② The liquid cladding metals will diffuse through the core metal.
③ The interlayer will have higher re-melting temperature than the bonding temperature.

A low cost and well-trusted bonding method at high temperature use

Objective

- The TLP bonding using Ni-Nb interlayer was successfully applied to bond HfB_2 composite in our previous work.(1)

- The adequate thickness of the interlayers of the joints is needed to explore.



Objective

The present work aimed at investigating the effect of Ni-Nb interlayer thickness on the mechanical properties of HfB_2 composite joints.

(1)Noritaka Saito:J.Mater.Sci, 47, 8454-8463(2012)

Experimental procedure- Fabrication of HfB_2 composites

Raw powders

HfB_2 (2 μm, 99.5%) + 10 vol% $MoSi_2$ (-2 μm, 99.9%)

Milling and Mixing

ZrO_2 milling media for 24 h in ethanol

Molding

Uniaxial pressing
+300 MPa CIPing

Sintering

1950℃ for 1 h
Under 0.1 MPa Ar

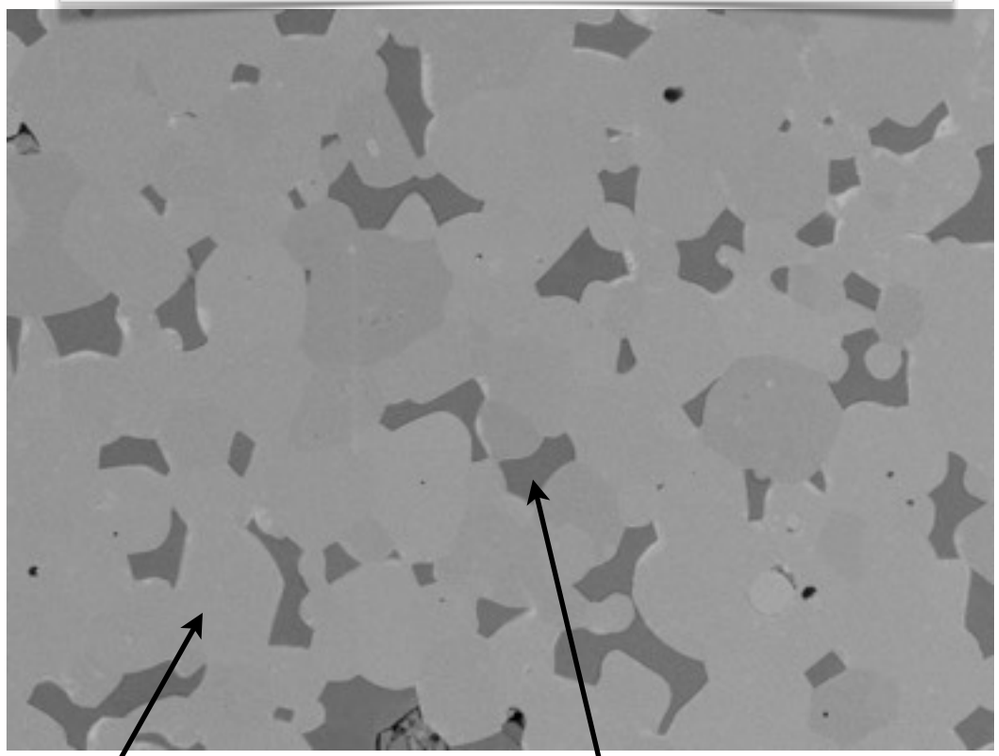
Polishing

with diamond slurry

Bonding

Relative density
97.6%

Microstructure of HfB_2 composite



Experimental procedure- Fabrication of HfB_2 composite joints

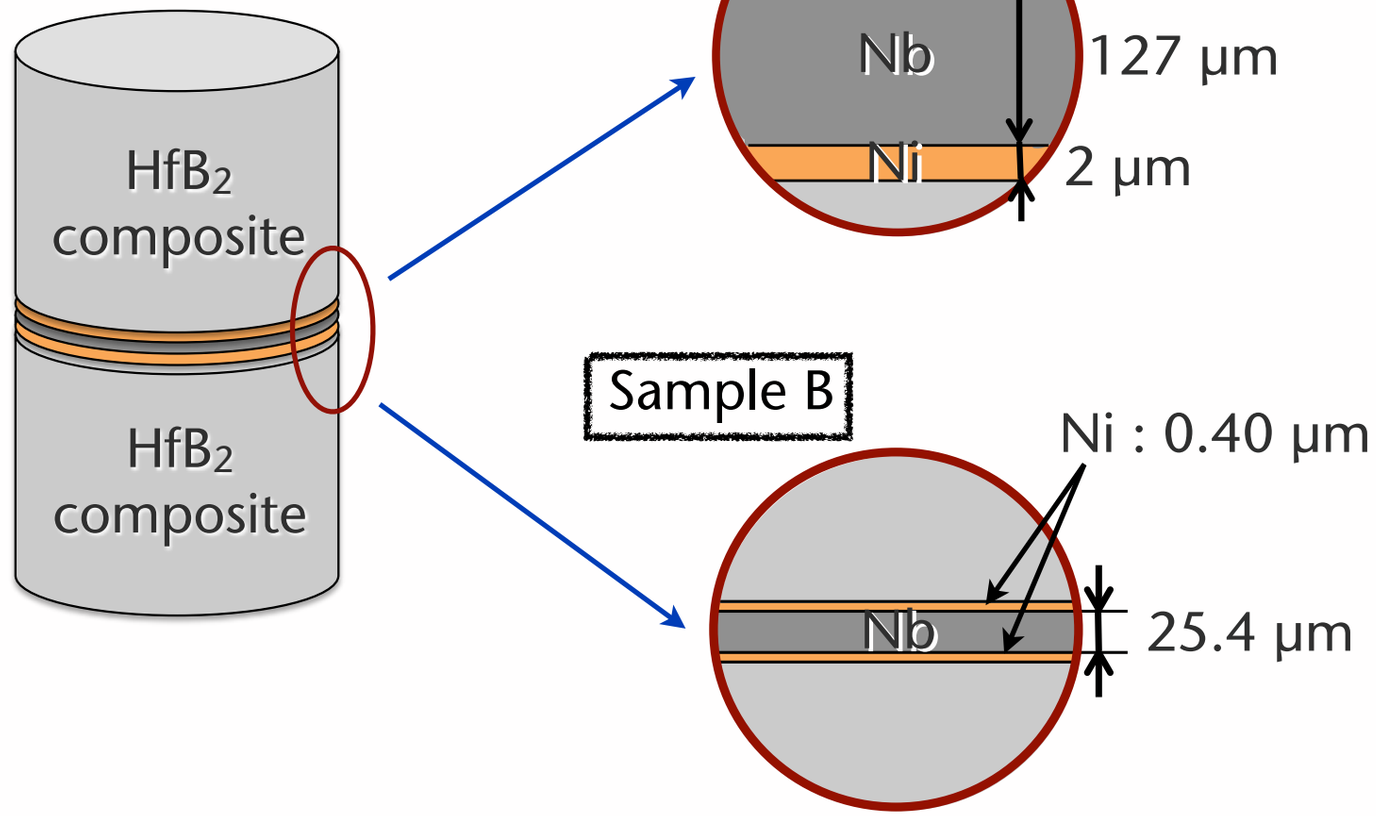
Interlayer

Cladding metal: Ni
Core metal: Nb

Hot pressing

Atmosphere: 20 Pa Vacuum
Temperature: 1500°C
Holding time: 30 min
Applied pressure: 8.5 MPa

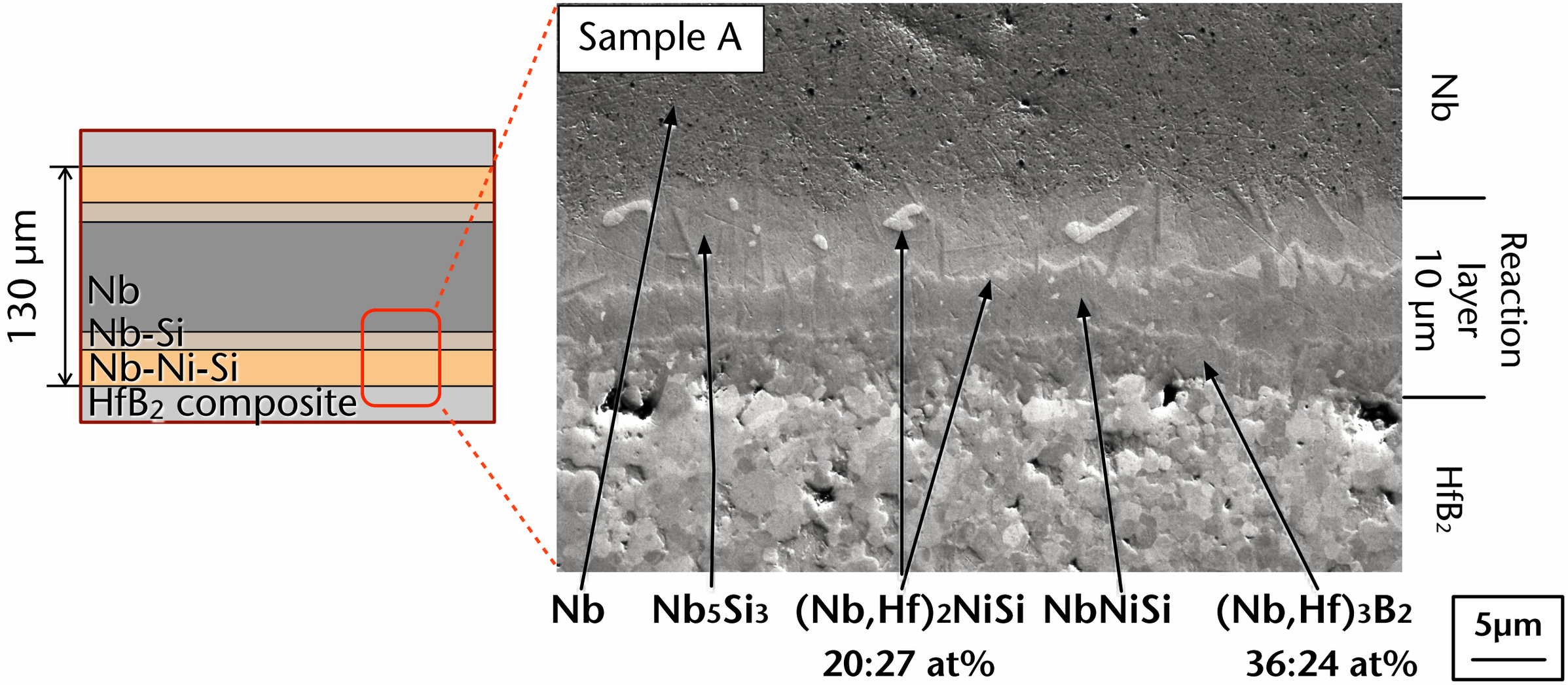
Quenching



- The interfacial region of joints were observed by using FE-SEM.
- The mechanical properties of joints were evaluated by 4-points bending test.

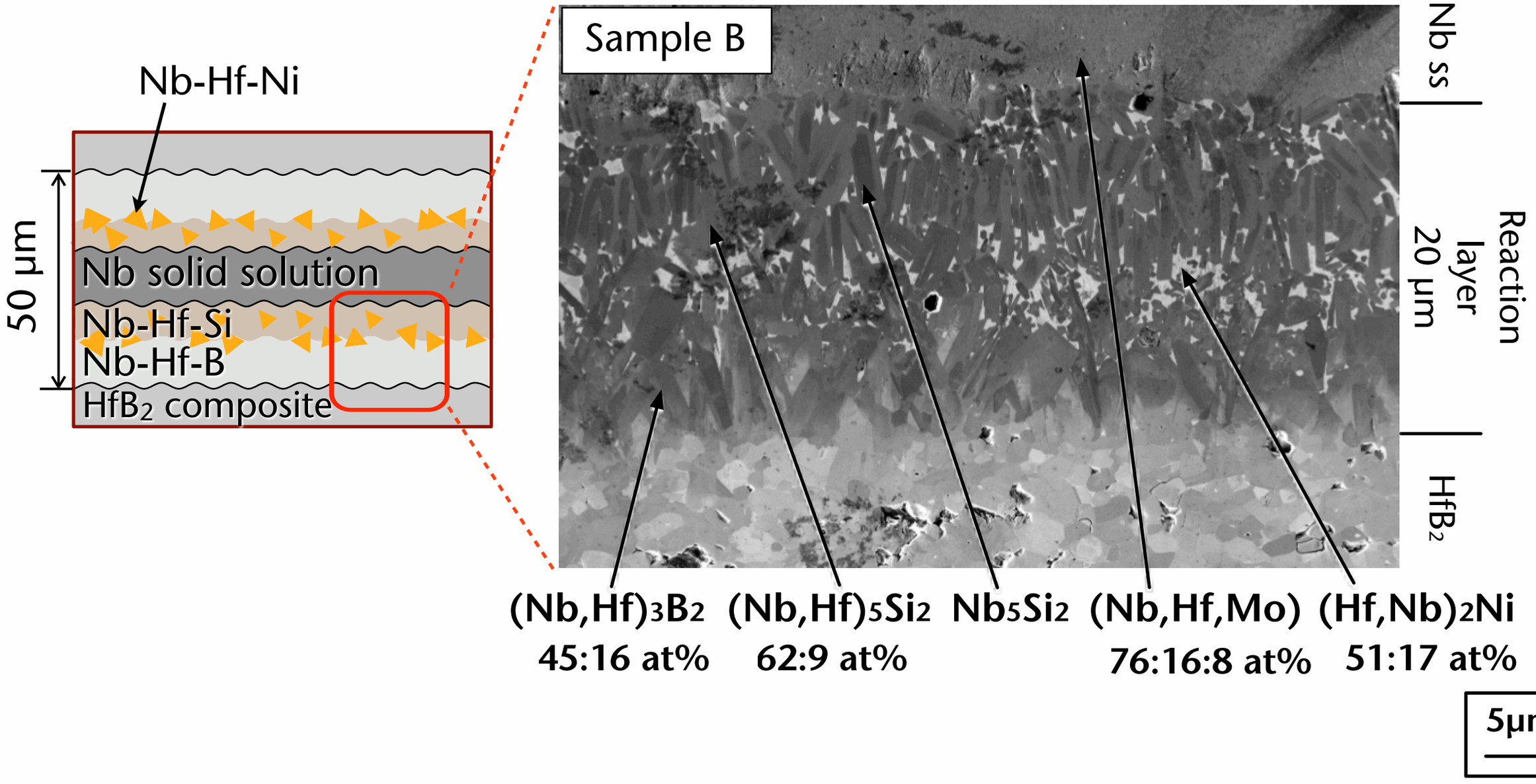
Observation results of interfacial reaction

Interfacial microimage of sample A (Nb : 127 μm, Ni : 2 μm)



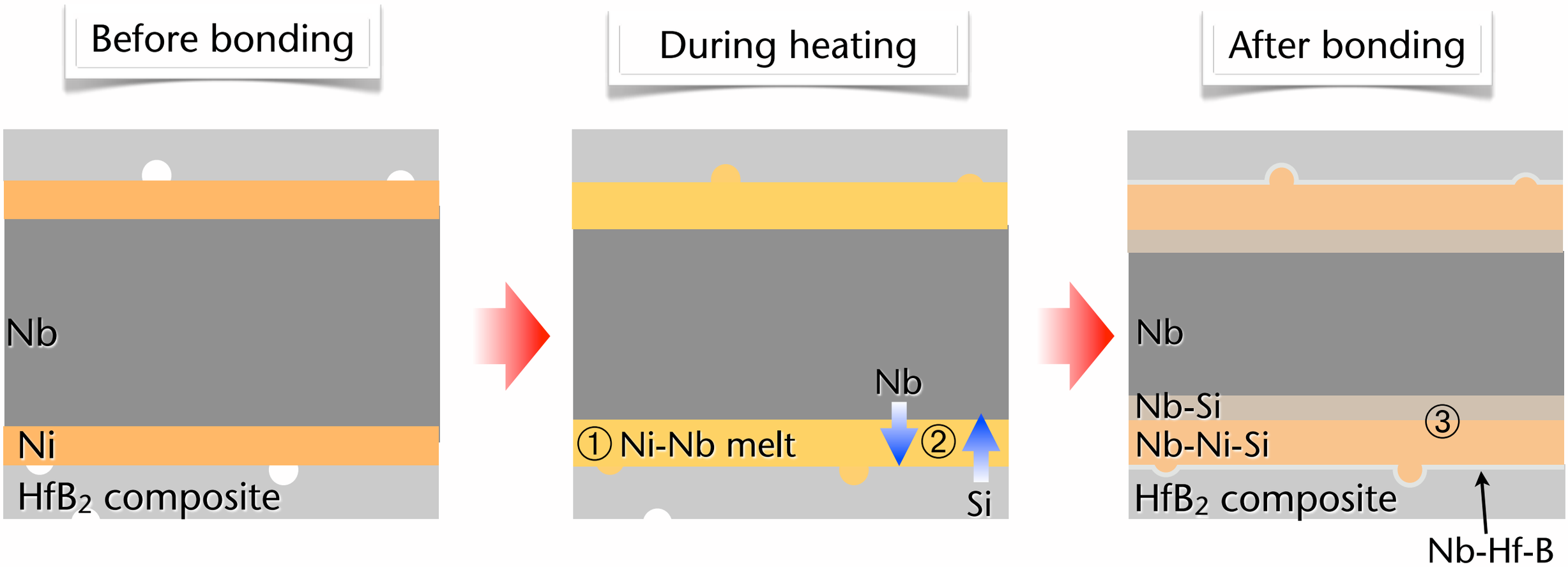
- The interdiffusion of Ni and Nb was not completed.
- The reaction layer mainly contained Si from $MoSi_2$ sintering aid.
- The HfB_2 composites hardly reacted to the interlayers.

Interfacial microimage of sample B (Nb : 25.4 μm, Ni : 0.40 μm)



- The interdiffusion of Ni and Nb was not completed similar to Sample A.
- The reaction layer contained Si and Hf from HfB_2 composite.

Discussion on the interfacial reaction of sample A

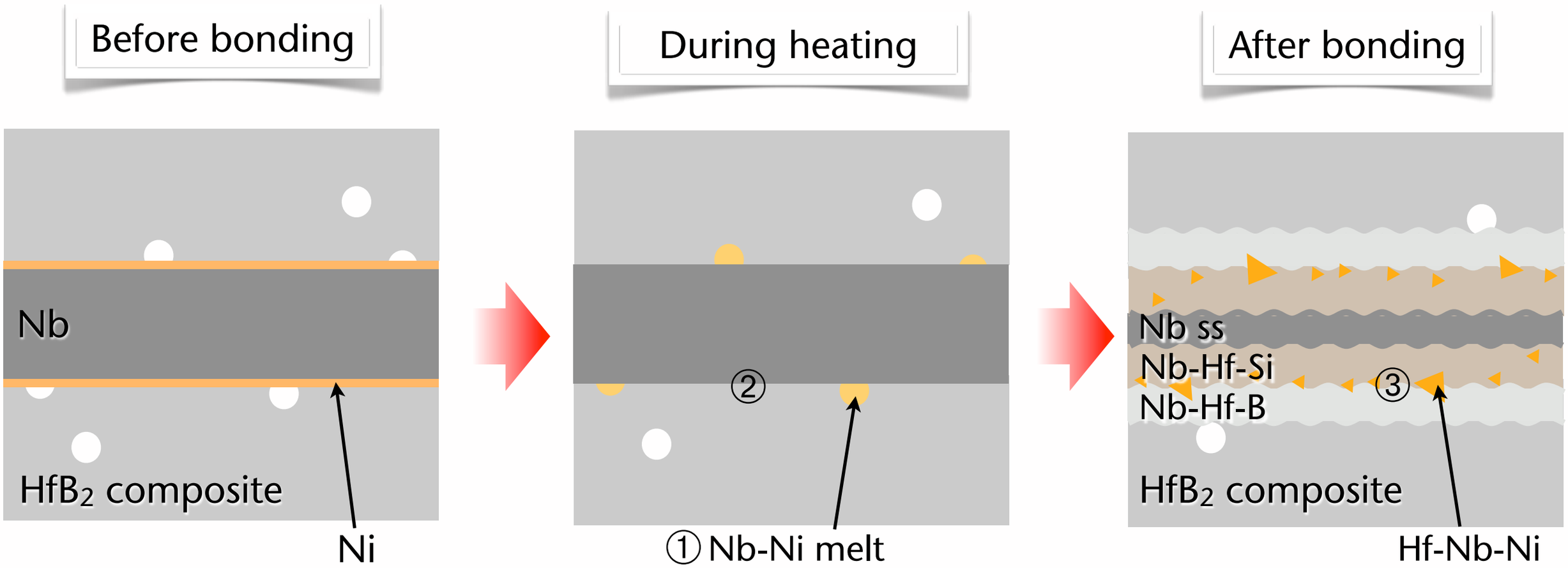


① Enough amount of Ni-Nb melt was formed, and filled the gap between Nb and HfB_2 composite.

② Nb and Si diffused into the other side respectively.

③ The thick reaction layer was formed uniformly.

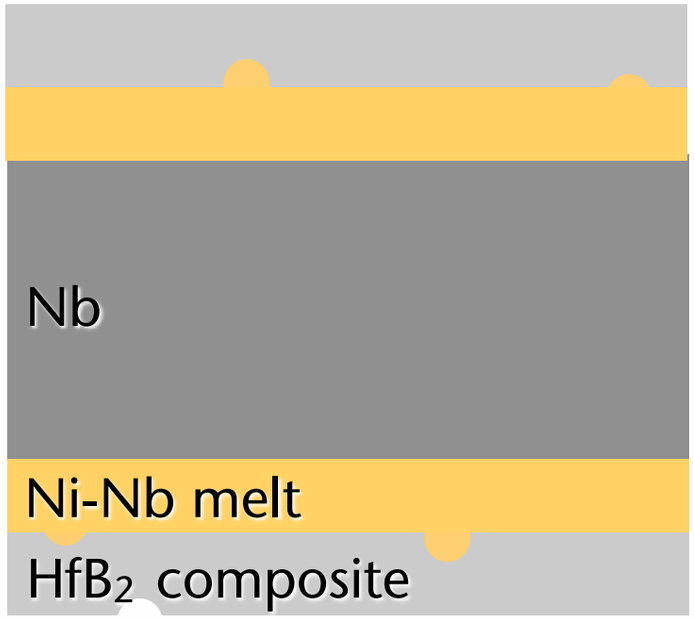
Discussion on the interfacial reaction of sample B



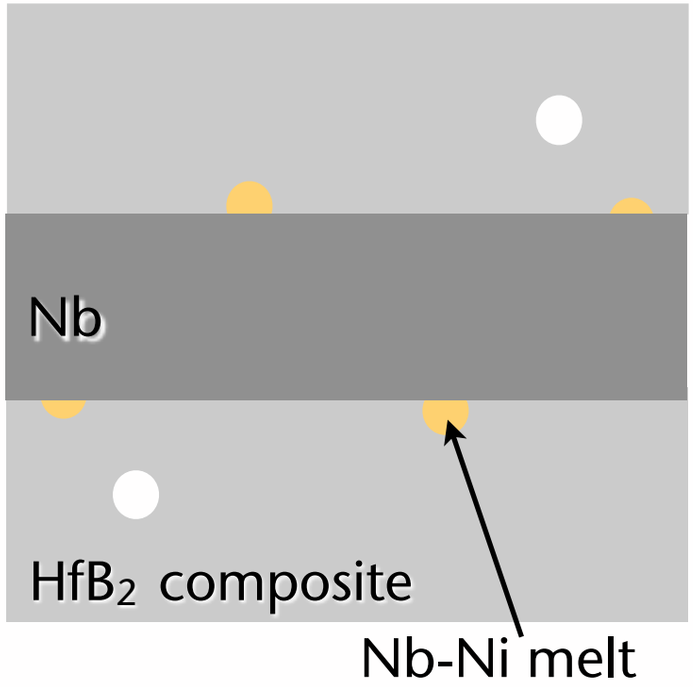
① Smaller amount of Ni-Nb melt was formed.
② Nb directly touched and reacted with HfB_2 composite.
③ The reaction layer was formed complexly.

Summary of interfacial reaction

Sample A



Sample B



Difference in the interfacial reaction is due to the difference in the amount of Ni-based melt.

4-points bending test

Fabrication process of bending beams for 4-points bending test

Bonding

Cutting

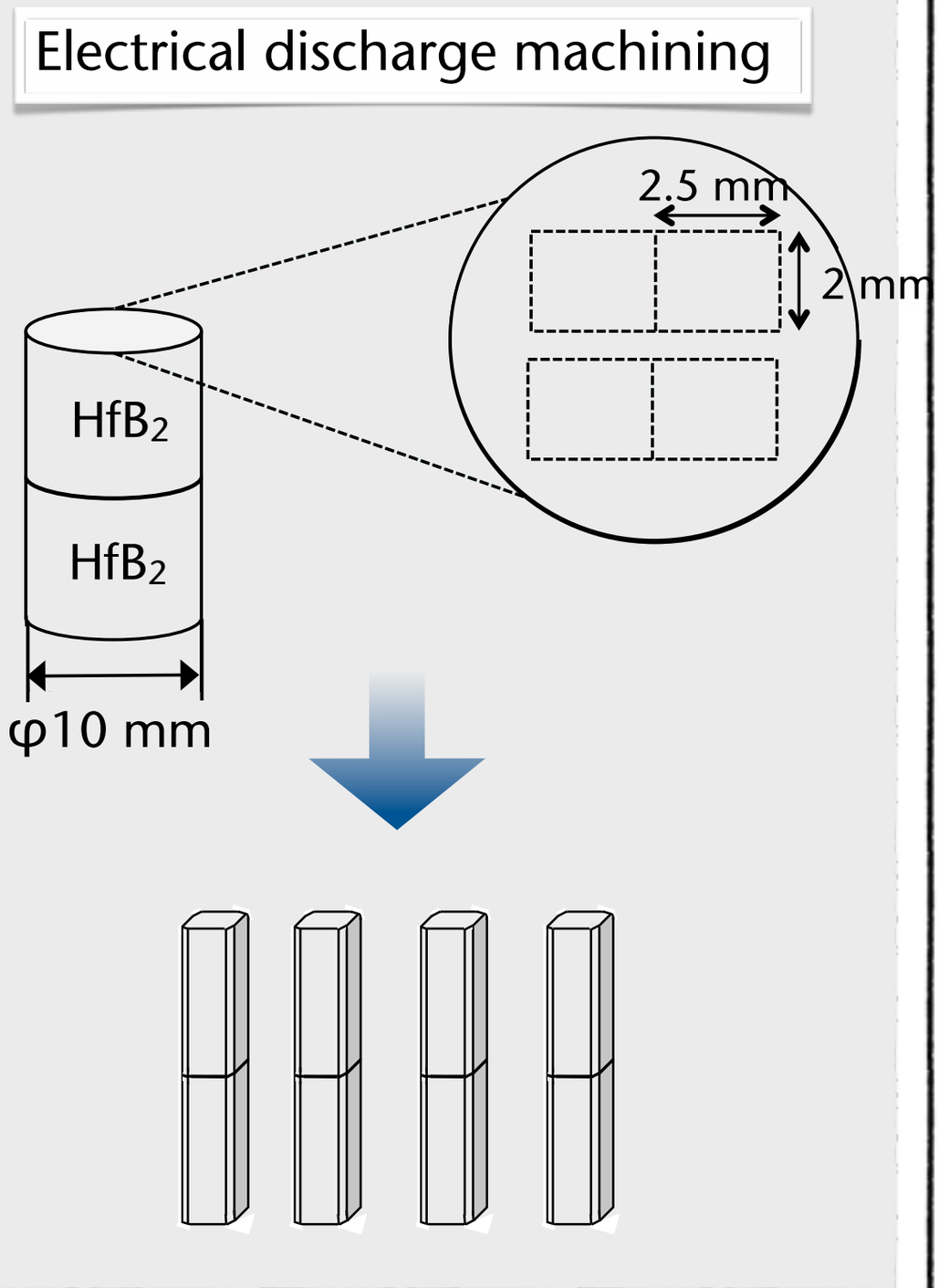
Electrical discharge machining
2 mm ×2.5 mm ×25 mm

Polishing

with diamond slurry

4-points bending test

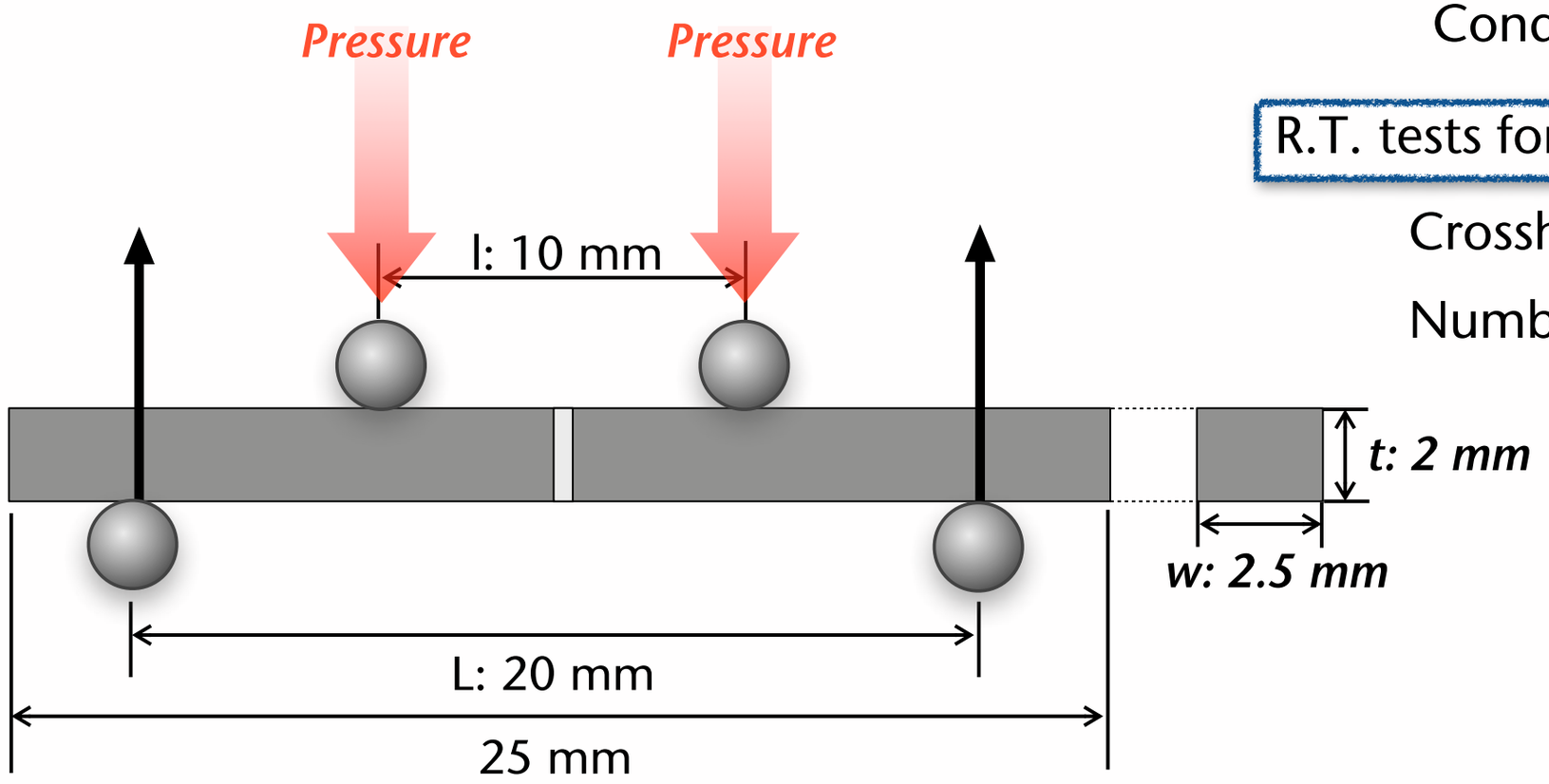
Strength measurement
at R.T. and at H.T. (1000℃)



Experimental procedure of 4-points bending test

$$\sigma = \frac{3P(L-l)}{2wt^2}$$

σ : Bending stress (MPa)
P : Maximum Load (N)
l : Loading span (mm)
L : Support span (mm)
w : Width (mm)
t : Thickness (mm)



R.T. tests for Joints

Crosshead speed : 1mm/min
Number of Trials : 5 times

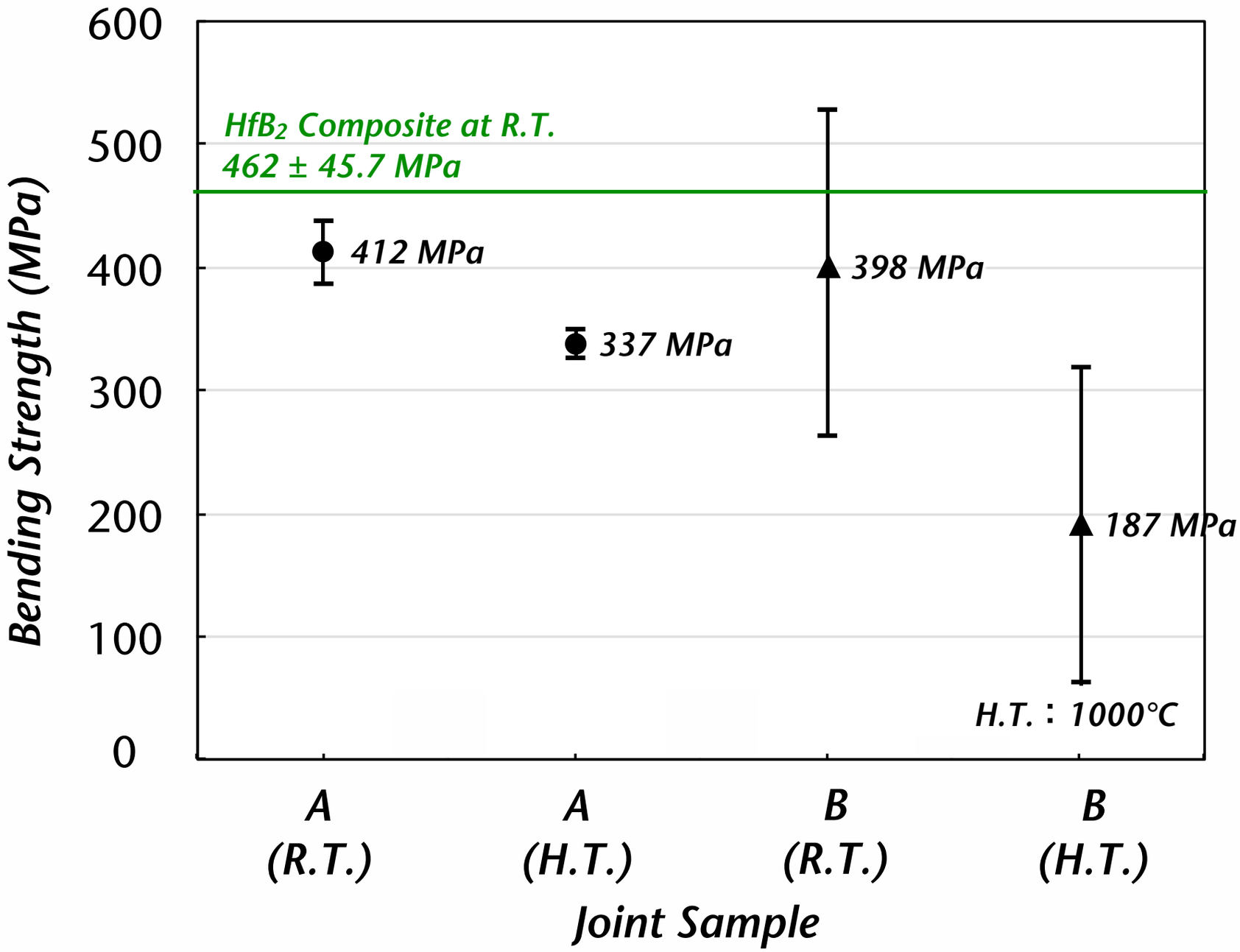
H.T. tests for Joints

Crosshead speed : 1mm/min
Number of Trials : 3 times
Conditions : 1000°C in air

R.T. tests for HfB_2 Composite

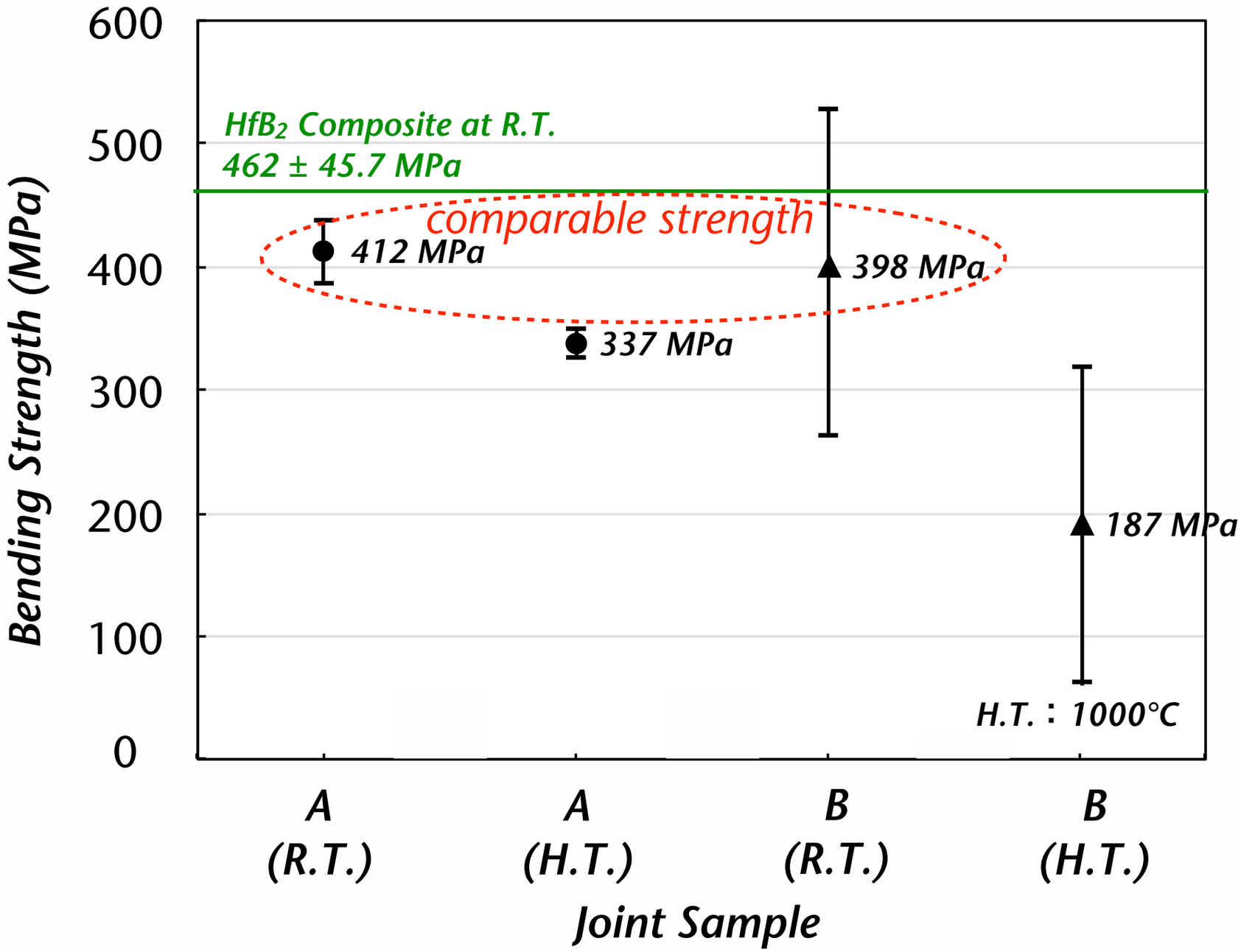
Crosshead speed : 1mm/min
Number of Trials : 12 times

Result and discussion of 4-points bending test



Result and discussion of 4-points bending test

SampleA and Sample B had similar strength, and had comparable strength with HfB_2 composite strength at R.T.

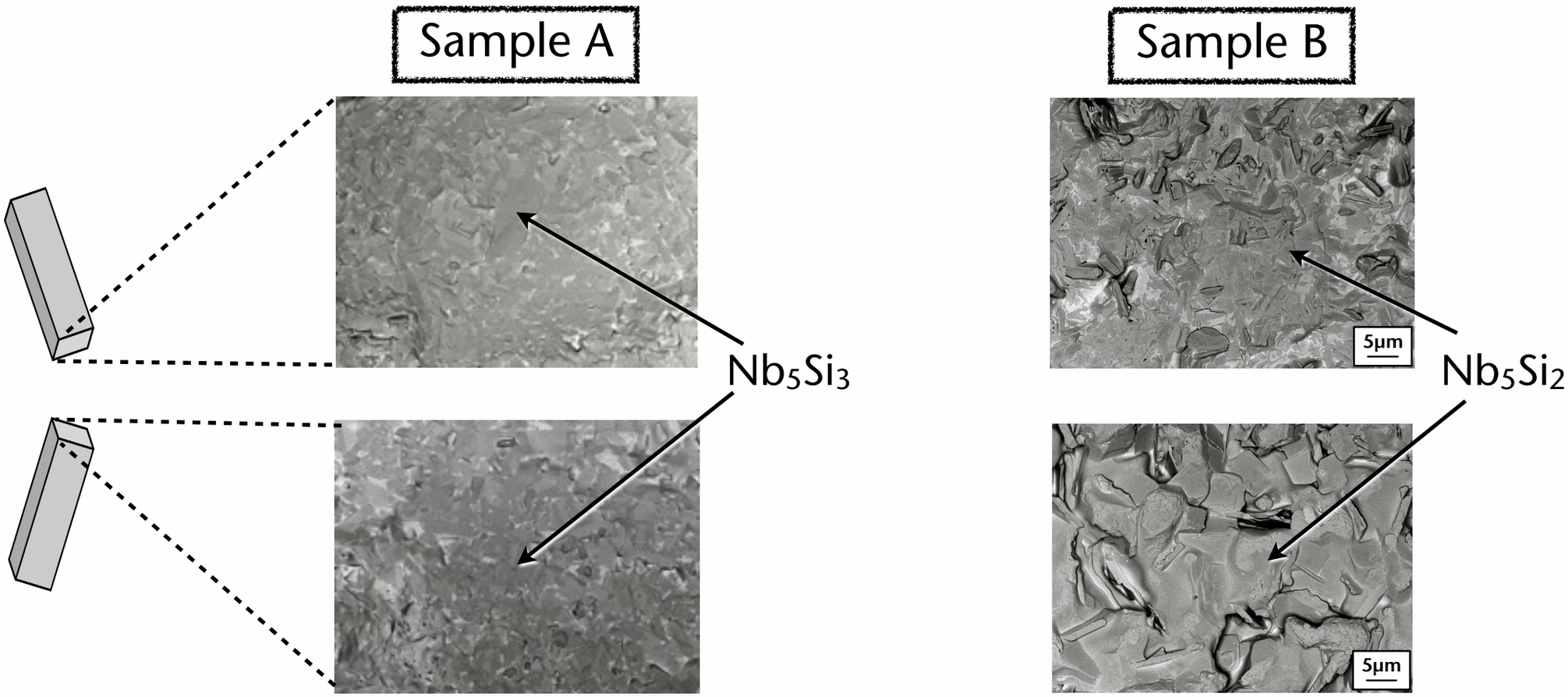


Result and discussion of 4-points bending test

SampleA and Sample B had similar strength, and had comparable strength with HfB_2 composite strength at R.T.

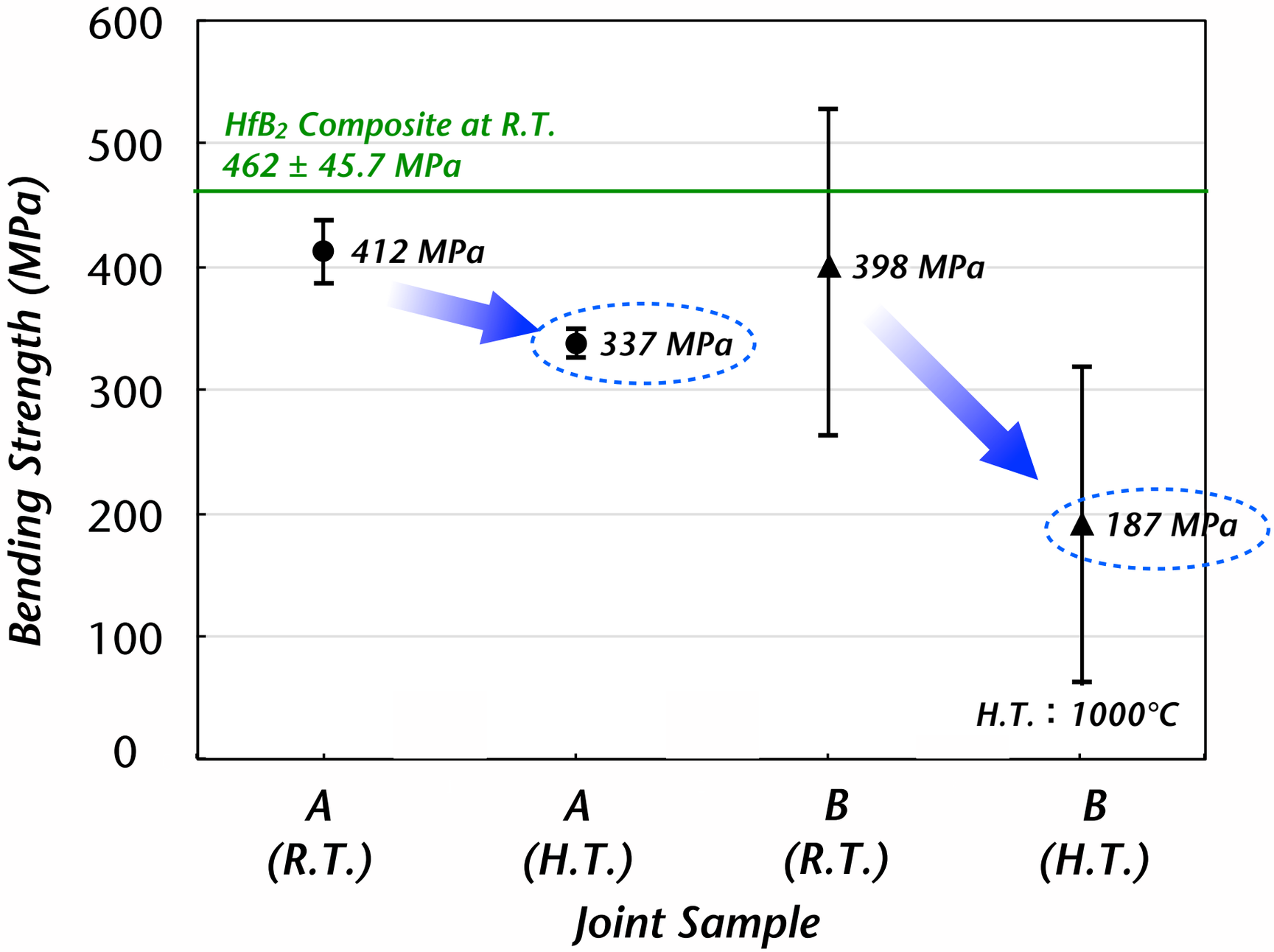
・From SEM observation of the fracture surfaces, similar Nb-Si were found.

・Ductile metal Nb would decrease the influence of residual stress in the cooling period of the bonding process.



Result and discussion of 4-points bending test

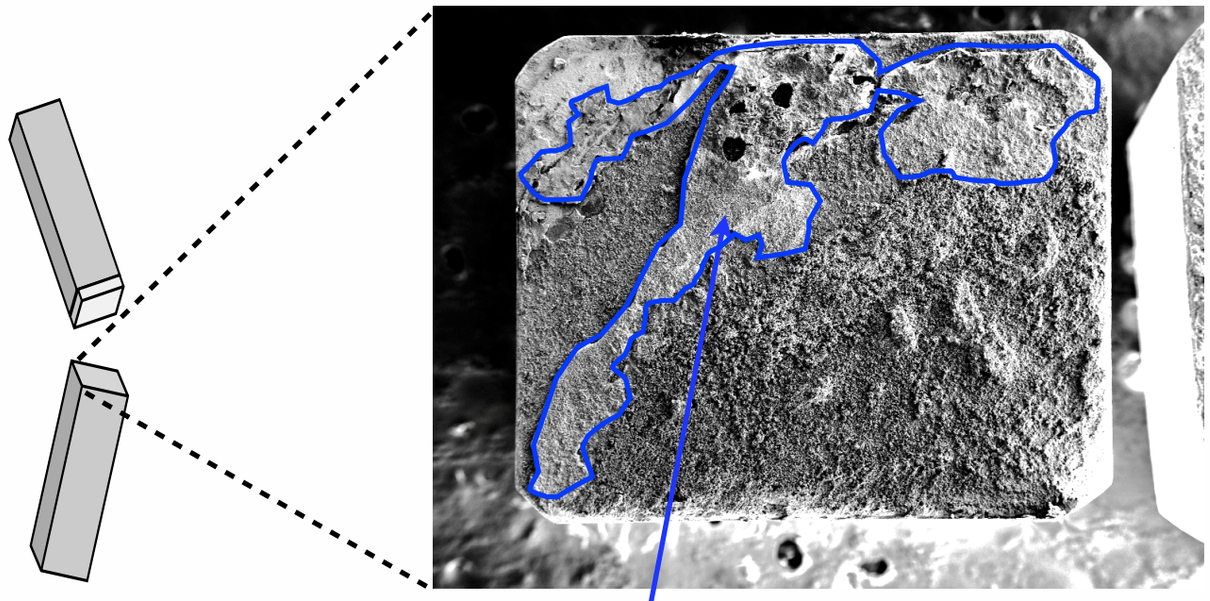
In Sample A, the H.T. strength was slightly decreased compared with the R.T. strength.
In Sample B, the H.T. strength was significantly decreased.



Result and discussion of 4-points bending test

In Sample A, the H.T. strength was slightly decreased compared with the R.T. strength.
In Sample B, the H.T. strength was significantly decreased.

- Some intermetallics phases of Ni-Nb were found.
- These Ni-Nb intermetallics have relatively low melting temperature.
- The presence and softening of these intrmetallics would be a possible reason why the H.T. strength of Sample B was found to be small.



Nb_7Ni_6+Nb_2Ni_6

Summary

The present work aimed at investigating the effect of Ni-Nb interlayer thickness on the mechanical properties of HfB_2 composite joints.

- HfB_2 composite joints were well-bonded by TLP bonding using Ni-Nb interlayer.
- Different thickness of Ni-Nb interlayer caused different reaction in the interfacial region.
- The different reaction was due to the difference in the amount of Ni-based melt.
- Two kinds of the joints revealed the similar strength at room temperature because similar Nb-Si was formed on the interfacial region regardless of the different reaction.
- Because Ni-Nb intermetallics have relatively low melting temperature, the intermetallics would significantly decrease the high temperature strength of the joints bonded with small amount of Ni.

Thank you !