



## DEPENDENCY OF MICROSTRUCTURE EVOLUTION AND MECHANICAL DURABILITY ON SINTERING TEMPERATURE OF W-SiC<sub>f</sub>/SiC<sub>m</sub> HYBRID COMPONENT

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### ABSTRACT

Reinforced silicon carbide matrix by silicon carbide nano fibers, hereafter SiC<sub>f</sub>/SiC<sub>m</sub>; is a promising material for nuclear fusion applications, due to the engineering aspects of the SiC<sub>f</sub>/SiC<sub>m</sub> materials; related to its limited thermal conductivity and durability, it was indispensable to make cladding of SiC<sub>f</sub>/SiC<sub>m</sub> substrate by higher thermally conductive, durable material such as Tungsten. This article discussed the conditions of the processing to produce durable joining between tungsten and SiC<sub>f</sub>/SiC<sub>m</sub> by hot pressing technique. The article discussed the correlation between these conditions and the microstructure evolution of the product at the fusion layer. The correlation between microstructure evolution and mechanical properties of the produced hybrid component of W- SiC<sub>f</sub>/SiC<sub>m</sub> was studied as well.

**KEY WORDS: NITE PROCESS, LHD, PLASMA FACING MATERIALS, W-SiC<sub>f</sub>/SiC<sub>m</sub> HYBRID COMPONENT.**

### 1- INTRODUCTION:

It is indispensable to conduct a special technique for solid state diffusion bonding between W and SiC<sub>f</sub>/SiC<sub>m</sub>, that purpose was achieved by nano infiltration transient eutectic (NITE process)[1-10] to produce hybrid component of W- SiC<sub>f</sub>/SiC<sub>m</sub>, this hybrid component showed a promising and attractive properties during its divertor plasma exposure test inside the large helical device(LHD)[11,12], the long term design aspect will take in consideration decreasing the thermal shocks resulting not only from quasi-steady state plasma, but also from the thermal load resulting from off-normal events[13].more precise investigation of the processing conditions of W- SiC<sub>f</sub>/SiC<sub>m</sub> and the dependency of mechanical and micro structure evolution on such processing condition is highly recommended aspect shall take on consideration; to reach to the optimum and sufficient data to produce durable bonded hybrid structure of W- SiC<sub>f</sub>/SiC<sub>m</sub>. This article showed the data of processing; five samples of W-SiC<sub>f</sub>/SiC<sub>m</sub> by hot press(HP) technology and studied the microstructure of three samples at their fusion bonding layer, the five samples were mechanically tested in destructive way; to show the correlation between the microstructure evolution with the mechanical test results

### 2- THE EXPERIMENTAL WORK

#### 2-1- preparation of the starting materials

The starting materials of the test samples are tungsten powder and SiC<sub>f</sub>/SiC<sub>m</sub> preform, where the dimensions of the preform is 40mm \* 40 mm\* 5.5 mm sheet has been fabricated as shown in Fig.1 with fibers of Tyranno<sup>TM</sup>-SA grade3, The preform is arranged as in Fig.1 inside the graphite mold where eventually the preform surrounded by graphite plates and

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carbon sheets to prevent sample to be stacked, the boron nitride sprayed between the graphite plates to facilitate releasing the sample after hot pressing process. The pure tungsten powder of purity 99.99% with rang size of 0.6 -0.99 μm with total weight 61.76 gm was piled and distributed above the preform

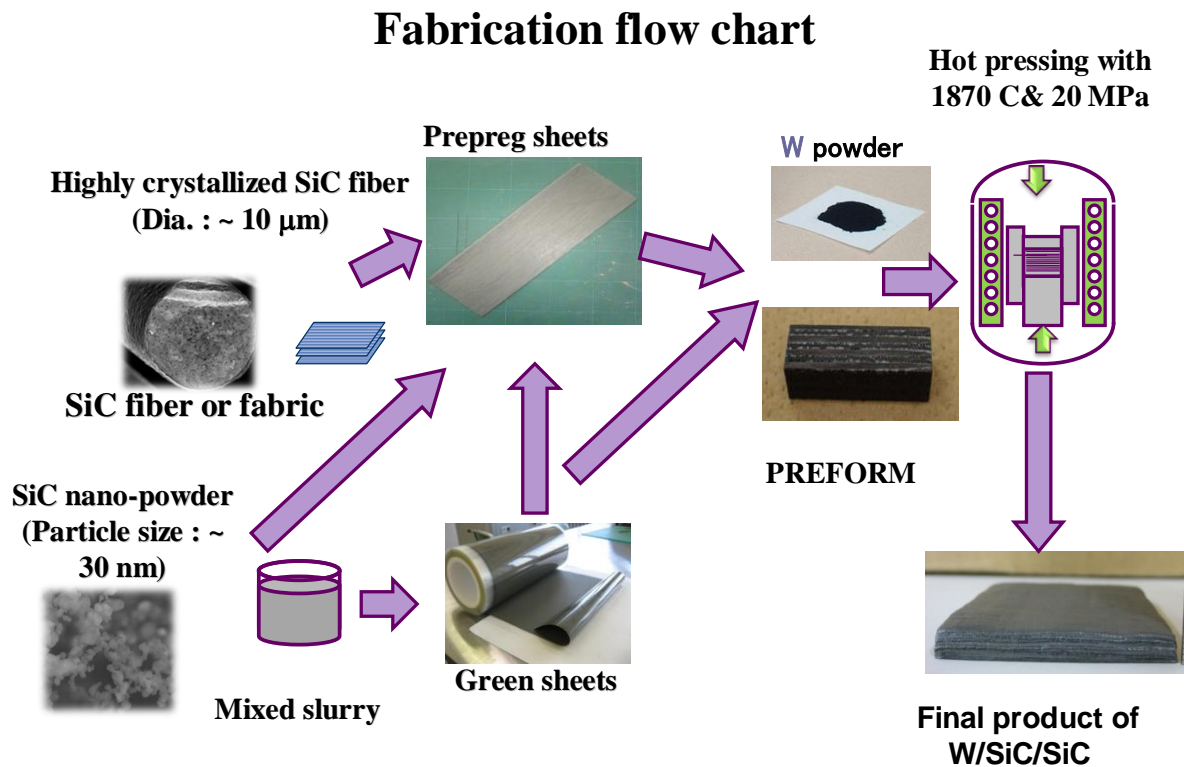


Fig.1. Flow chart of the original NITE process

### 2-2 Hot -press operating condition

The hot pressing operating conditions representing on the temperature and applied pressure; The trial samples were fabricated with temperatures are ranging from 1700°C to 1900°C, where five trial samples were fabricated; using temperatures; 1700 °C, 1750 °C, 1800 °C, 1850 °C 1900°C, all these samples were subjected to a constant heatin up rate up to it is maximum degree, then each sample was holded atit is maximum temperatur for one hour, during heating up of each sample a synchronized pressure was applied with a constant rate, till maximum pressure up to 20Mpa, then the hot press holded the applied pressure for one hour [14]. the conditions of each sample are illustrating in table (1)

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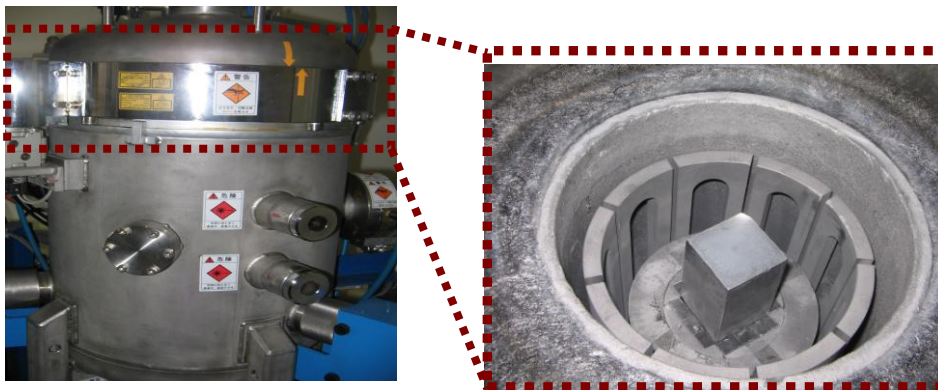
**Table (1). Specification of the hot press machine**

Sample No.	Applied temperature(°C)	Holding temperature time(minutes)	Applied pressure(MPa)	Holding pressure time(minutes)
1	1700	60	20	60
2	1750	60	20	60
3	1800	60	20	60
4	1850	60	20	60
5	1900	60	20	60

Hot pressing machine is shown in **Fig.2** - model (FVPH-P-R-5FRET-15), the Specification of the hot press machine is illustrated in **table (2)**, the graphite mold of the hot press was used for fabrication the starting materials as illustrating in **Fig.1**.

**Table (2). Specification of the hot press machine**

Model	Max temperature	Heating time	Mold dimensions	Vacuum	Atmosphere pressure	Press pressure	Electric power
<b>FVPH P-R-5FRE T-155</b>	1900°C	40 minute R.T to 1500 °c	Φ 120 mm H 110 mm	1*10 <sup>-4</sup> pa	0.92 M.pa N <sub>2</sub> , Ar	4.9 *10 <sup>4</sup> N	220 V 15 KW

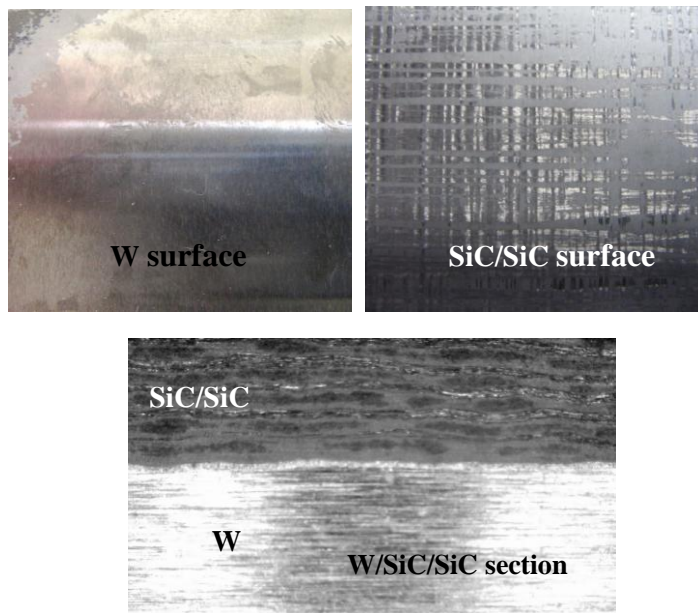


**Fig.2. Hot press machine and its inner mold**

### 2-3-Samples take out and preparation

After finishing the sintering by hot press of each sample according to the above mentioned conditions, the five samples; Nos: 1, 2, 3, 4, 5 were taken out and prepared for subsequent tests, where taken out samples were polished and grinded by rotation polishing technique (LOGITEC PM5) to the smooth surface and texture of both side of tungsten and SiC<sub>F</sub>/SiC<sub>M</sub>, as illustrated in the Fig.3.

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**Fig.3. test sample showed the surface of both SiC<sub>f</sub>/SiC<sub>m</sub> and W and cross section**

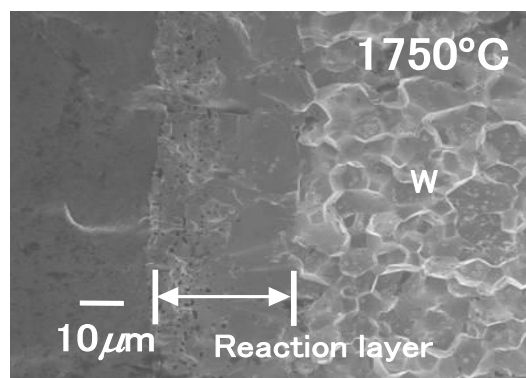
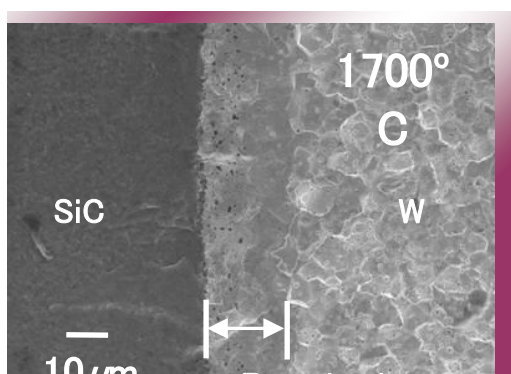
**3-RESULTS AND DISCUSSION**

**3-1Microstructure evolution**

All test samples were examined at the cross section of tungsten- SiC<sub>f</sub>/SiC<sub>m</sub> interphase layer, to investigate the microstructure evolution; on the reaction layer between tungsten and SiC<sub>f</sub>/SiC<sub>m</sub>, the microstructure investigation showed that; there was a phenomenal change in the interphase layer thickness, due to different processing conditions of sintering temperature. The relation between sintering temperature and the thickness of the reaction layer is illustrated in table.3. where at sintering temperature of sample "1" at 1700°C; showed reaction layer thickness up to 25 μm, whereas the samples "2","3","4","5" with sintering temperatures: 1750°C, 1800°C,1850°C,1900°C, showed a thicknesses of reaction layer:33,40,45,78 μm respectively. Fig.4. showed the microstructure evolution of the reaction layer of samples "1","2","3","4","5", where slightly increase in thickness of reaction layer was observed in samples;1,2,3,4, whereas high accelerated increase was demonstrated in sample 5

**Table(3). the correlation between sintering temperature and thickness of reaction layer**

Sample No	Sintering Temperature(°C)	Thickness of the reaction layer(μm)	comment
1	1700	28	Slight increase
2	1750	33	Slight increase
3	1800	40	Slight increase
4	1850	45	Slight increase
5	1900	78	Accelerated growth



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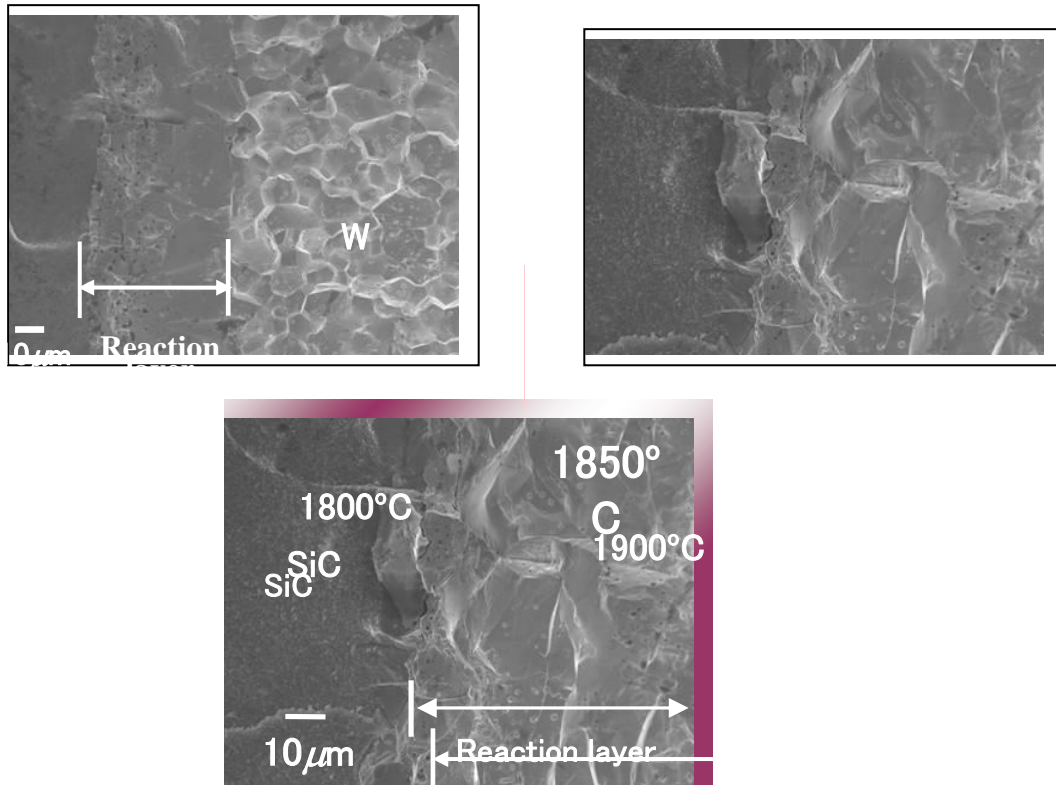


Fig.4. the microstructure evolution of the reaction layer of three samples, with different sintering temperatures

### 3-2 Flexural strength test

Five hot pressed samples were tested mechanically by three point bending test, where the five samples; with different sintering temperature "1"(1700°C) "2"(1750°C),"3"(1800°C),"4"(1850°C),"5"(1900°C) was bended by the device of 5581, Instron Inc with a cross head speed: 0.5mm/min, specimen size: 22×2×2 [mm] as shown in the Fig.5. The results of the flexural strength of the bended samples are shown in Fig.6, where, the correlation between sintering temperature and value of flexural strength behaved as proportional linear function, where the values of flexural strength at 1700°C,1750°C,.1800°C, were 680,1050,1150 Mpa respectively,on the other hand when the sintering temperature commenced to be elevated after 1800°C, it showed non-proportional linear function correlation with the values of flexural strength, where after 1800°C to 1850°C showed slight deterioration of the flexural strength values from 1150 MPa to 1050 MPa, after 1850°C to 1900°C showed catastrophic deterioration in flexural strength from 1050 to 500 Mpa.

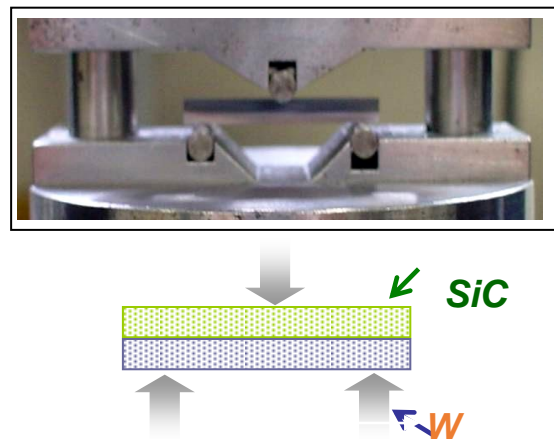
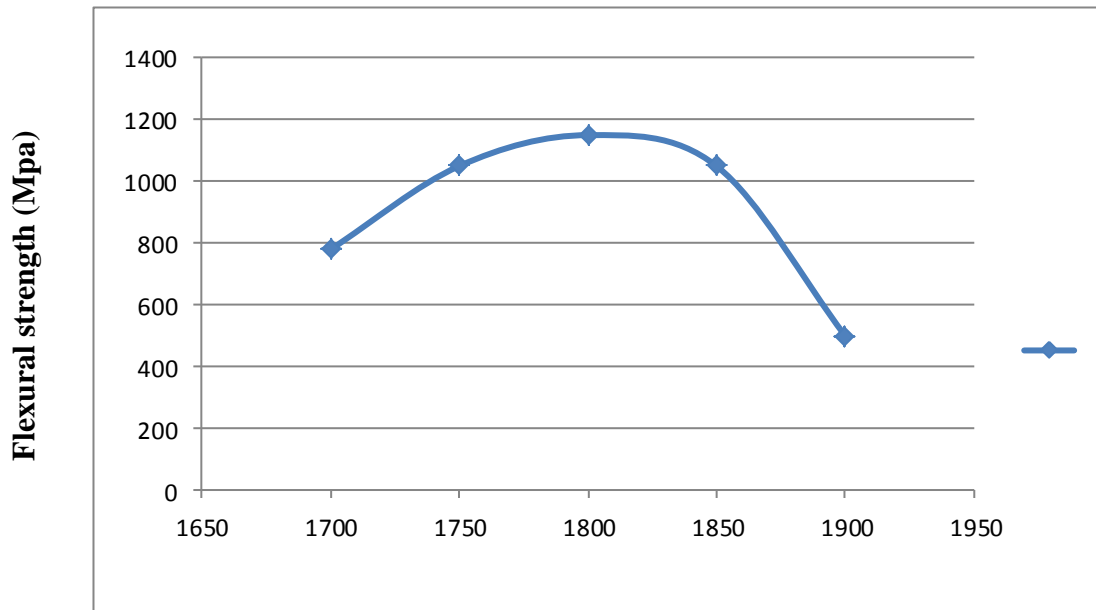


Fig. 5. device of three point bending test

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**Fig.6. Correlation between sintering temperature and Flexural strength.**

**3-3-Chemical analysis of the interphase layer and its relation with the flexural strength .**

Each processed sample was tested by XRD method in the region of the reaction layer, to make the correlation between the chemical phases appeared on the reaction layer and the mechanical test results of the flexural strength, where the results of XRD due to the analyses of each SEM images(Fig.7);are tabulated on the table.(4) the chemical analyses, could clearly interpretate the behavior of the flexural strength of each sample where as seen in table(4) the samples;1,2, with fabrication temperatures;1700°C, 1750°C respectively, showed the occurrence of phases; W<sub>2</sub>C, WC, WSi<sub>2</sub>,wherease the samples 3,4,5 with fabrication temperature; 1800°C, 1850 °C, 1900°C respectively, showed a disappearing of the phase(WSi<sub>2</sub>), and domination of the phase (W<sub>5</sub>Si<sub>3</sub>), which it's astrong evidence that the deterioration of the flexural strength after temperature 1800°C was due to the domination of the phase W<sub>5</sub>Si<sub>3</sub> and disappearing of the phase (WSi<sub>2</sub>).

**Table (4). X-Ray analysis of the reaction layer at different sintering temperatures**

Sintering temperature(°C)	XRD phases appeared in the interphase layer		
<b>1700</b>	<b>W<sub>2</sub>C</b>	<b>WC</b>	<b>WSi<sub>2</sub></b>
<b>1750</b>	<b>W<sub>2</sub>C</b>	<b>WC</b>	<b>WSi<sub>2</sub></b>
<b>1800</b>	<b>W<sub>2</sub>C</b>	<b>W<sub>5</sub>Si<sub>3</sub></b>	<b>WC</b>
<b>1850</b>	<b>W<sub>2</sub>C</b>	<b>W<sub>5</sub>Si<sub>3</sub></b>	<b>WC</b>
<b>1900</b>	<b>W<sub>2</sub>C</b>	<b>W<sub>5</sub>Si<sub>3</sub></b>	<b>WC</b>

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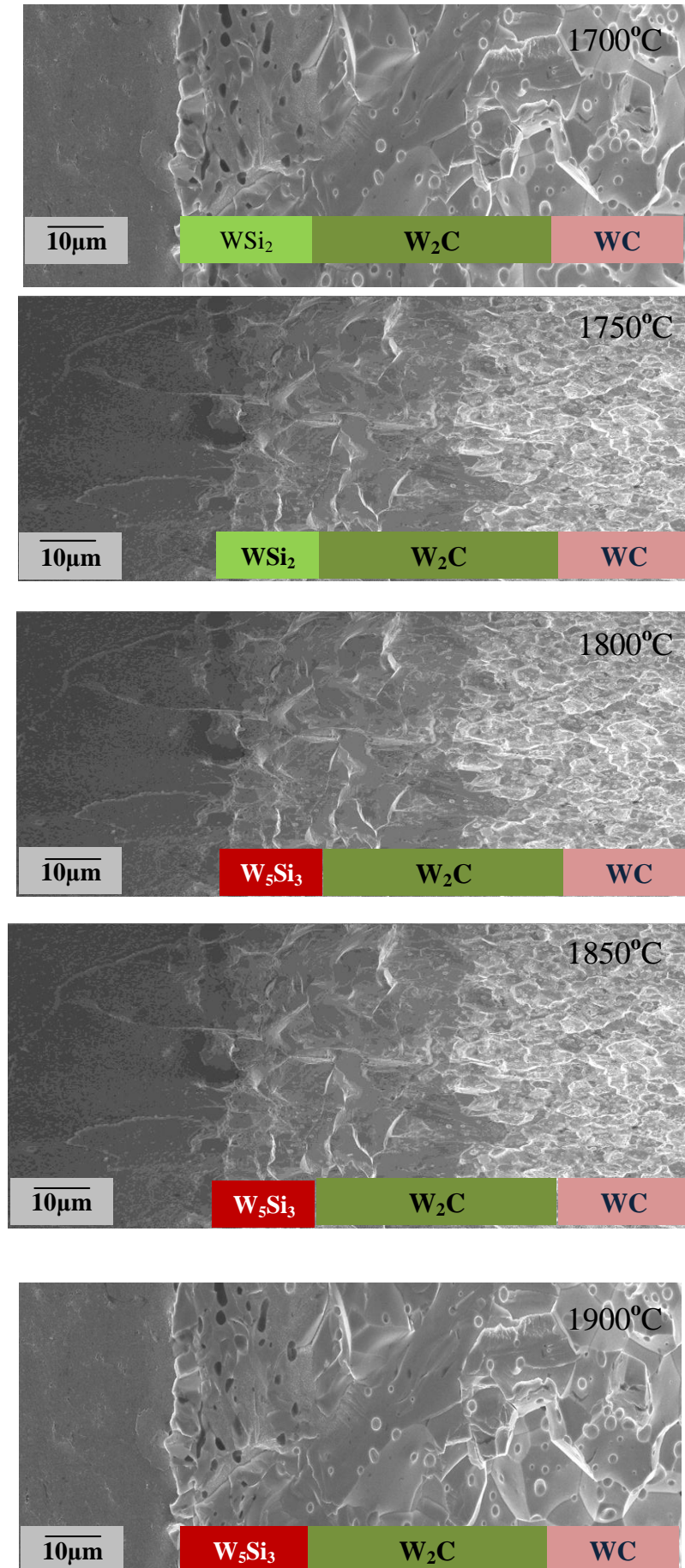


Fig.7. SEM images showing XRD of reaction layer of three samples of different sintering

#### 4-CONCLUSION

The results of the analyzed data showed that; the durability of the W- SiC<sub>F</sub>/SiC<sub>M</sub> hybrid component doesn't depend only on the thickness of the reaction layer, due to increasing the sintering temperature, but it depends also on the chemical composition of the formed reaction layer in the interphase region, where the optimum sintering temperature which showed threshold of the maximum flexural strength, up to 1150 Mpa at 1800°C, due to the substitution of the phase "WSi<sub>2</sub>" by the phase "W<sub>5</sub>Si<sub>3</sub>", the last phase dominated with increasing the sintering temperature, which resulting to the catastrophic deterioration of the mechanical flexural strength at the temperature of 1900°C, from 1050Mpa to 500 MPa, due to the domination of the phase "W<sub>5</sub>Si<sub>3</sub>". According to the resulting data; it is strongly recommended to conduct the sintering at the temperature 1800°C at pressure 20MPa for holding time 1hr.

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