Fabrication and superplastic forming property research of TiB whisker reinforced titanium alloy matrix composite sheet

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Abstract. Applied at elevated temperature compared with in situ-whisker reinforced titanium matrix composite(TMC) higher than matrix titanium alloy with 50~150℃. The advantage of TMC compared with Ti-Al intermetallic compound is lower cost, better weldable property, simple fabrication process; lower density compared with super alloy. One of important demand of TMC sheet for high temperature application is used for higher speed craft with lighter part fabricated by SPF/DB technology. This paper illustrated TiBw/TA15+ TMC sheet fabrication and SPF/DB processing property, and analyzed effects of rolling processing on the microstructure and tensile properties. Wide sheet has been fabricated successfully. Forming mechanism of TMC is revealed, which is different from conventional titanium alloy, fined microstructure is unnecessary for TMC, four layers SPF/DB component also can be formed for TMC sheet.

1. Introduction

More than 40 years passed from the first 600°C high temperature titanium alloy(IMI834) successfully developed, because decrease capability of the anti-oxidation and stability of the microstructure, then application at elevate temperature no beyond 600°C applied in the aero engine. The focus research on high temperature low density alloy turn to titanium-aluminium intermetallic compound. However, the extreme brittleness and lower fracture toughness still not very satisfied the requirement of engine part, including manufacture suitability, just a few parts was successful applied. Traditionally, strengthen way for high temperature titanium alloy at high temperature are solution, α2 phase and silicides. the in situ titanium matrix composite could keep increasing the strength at higher temperature than matrix through add another one or two second phase as TiB or TiC, usually 50~150°C higher, and temperature and mechanical properties could be optimized of in situ TMC, which is a very prospective high temperature structural material.

In this paper, Si and B are added into Ti-6.5Al-2Zr-1Mo-1V alloy to fabricate the in situ TMC sheet, and further investigate the SPF/DB combined processing experiments; the four layers structure part is successfully developed.

2. Experimental

The in situ TMC reinforced with 3.5 vol.% TiB/ Ti-6.5Al-2Zr-1Mo-1V cast ingot was prepared by vaccum arc remleting(VAR), and totally remelted three times. Several billet sections were cross worked by multiple upset and redraw operations at β single field and α+β phase field with a total 9 times reheating and working, to create a more homogeneous billet structure and higher ratio of TiB whiskers broken as much as possible, then billet is rolled on a 1200mm wide roller machine at preheating temperature of β_T-20°C, β_T+50°C, β_T+100°C respectively, and finally the thickness of 1.2mm and 1000mm wideness sheet is fabricated with the same surface quality as matrix alloy, no defect was found by ultrasonic inspection. Annealing treatment applied at 860~940°C/1h AC.

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Superplasticity and diffusion bonding processes is examined with 900~960℃ with uniaxial superplastic tensile and gas pressure diffusion bonding respectively. The optimized parameters are used to form a four layers SPF/DB combined structure.

Metallographic preparation through grinding and polishing, followed by immersion etching in Kroll's reagent. Microstructure was studied using ZEISS optical micrograph and SEM.

3. Results and discussion

3.1 Rolling temperature effects on microstructure and tensile property

Hot rolling operation is done at 20℃ below β transus, the microstructure is combined with primary α and β transformed with a-laths, and primary α distributed align to the rolling direction, the formed micro voids due to the long TiB whiskers fracture without filling with matrix. With rolling temperature increasing, microstructure turn to be complete β transformed with α-laths. Distribution of TiB whiskers is related homogeneity, but few micro voids still can be found. Furthermore, the micro voids decrease obviously with increasing rolling temperature, as shown in Fig. 1.

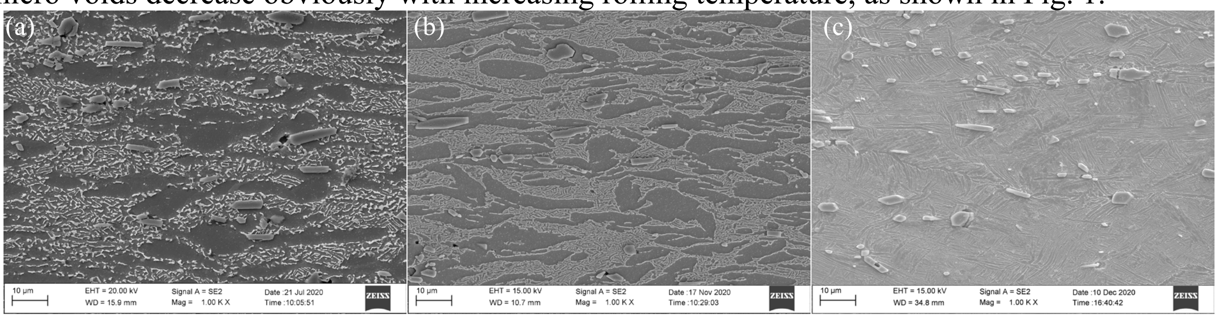

Fig.1 Microstructures of TMC sheet rolled under different temperatures
(a) β_T-20 ℃, (b) β_T+50 ℃, (c) β_T+100 ℃

Fig.2 shows tensile properties results at RM and 650℃, better RT ductility and middle high temperature ultra tensile strength are obtained which rolled at 20℃ below β transus, the average is 7.8% and 457MPa respectively. However, the highest average of UTS is 523MPa at 650℃ which is rolled at 100℃ above β transus. For the whisker reinforced TMC, the ductility at room temperature is one of key technical difficulties. Above results show micro or nano dimension of TiB whiskers could improve the ductility at RT.

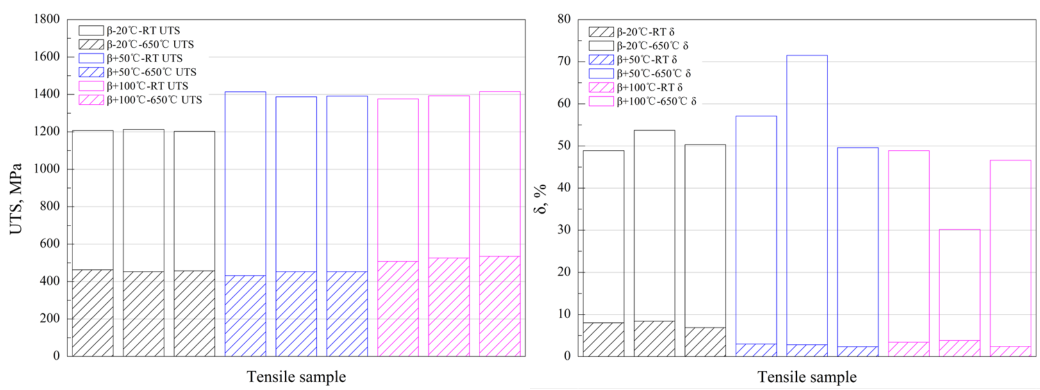

Fig.2 Tensile properties at RT and 650℃ of rolled condition (a) UTS (b) δ

2.2 Effects of annealing temperature on microstructure and tensile properties

Based on the sheet rolled at β_T-20℃, compared with microstructure of rolled sheet, after 860℃, 900℃ and 940℃ annealing for 1h, the obviously changes happened in second precipitation α-laths, it becomes wider and even being to equiaxed, and contents of α-laths decrease with increasing anneal temperature. However, TiB whisker is very stable, as shown in Fig. 3.

Fig. 4 shows tensile results at RT and 650℃, 900℃/1h is the optimized anneal parameters according to balance of 650℃ strength and RT ductility. Tensile ductility at RT and ultra strength at 650℃ are 6.9% and 580MPa respectively.

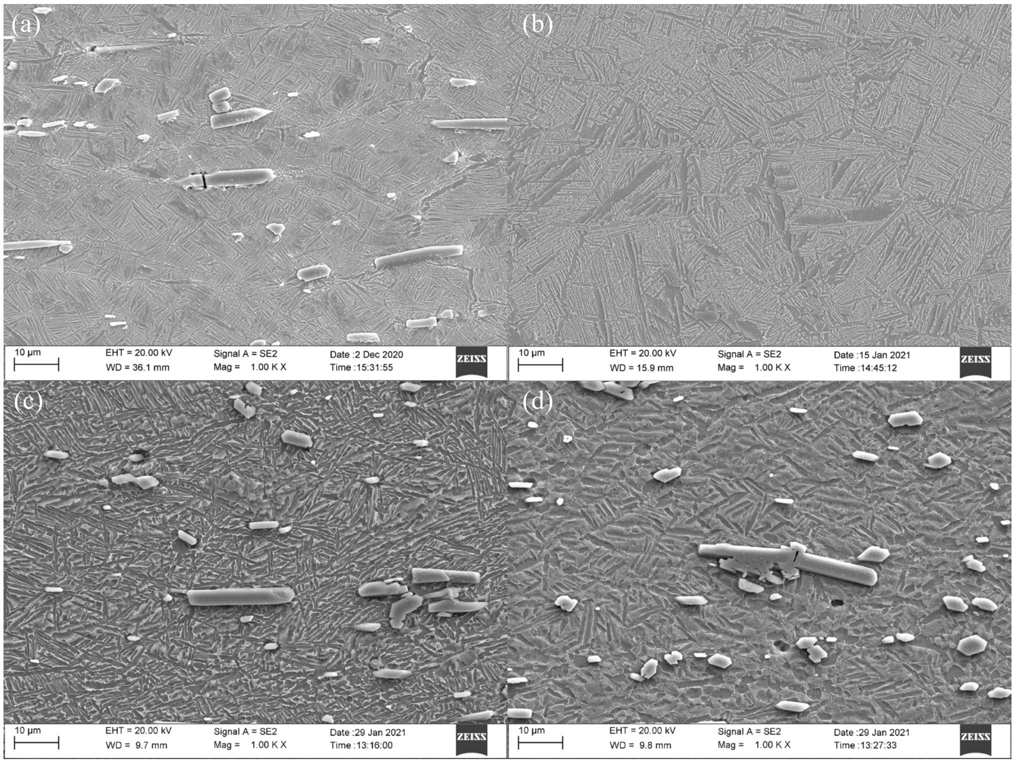

Fig.3 Microstructures after anneal (a)rolled; (b) 860 ℃/1h, (c) 900 ℃/1h, (d) 940 ℃/1h

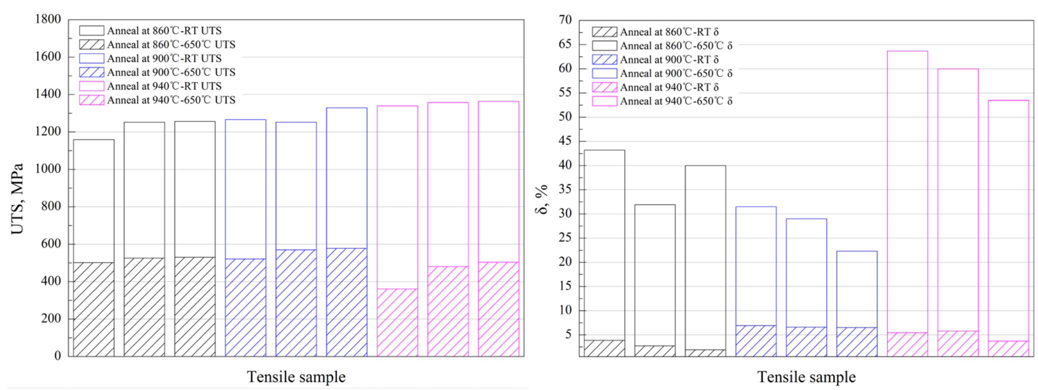

Fig.4 Tensile properties at RT and 650 ℃ after annealing at different temperature (a) UTS (b) δ

2.3 Superplasticity evaluation

According our previous and present researches, this article combined the results of uniaxial superplastic tensiles. Compared superplastic tensile results with 5.0 vol.% and 3.5 vol.% TiB whisker content, the best ductility of 569% obtained at 960℃、$1\times10^{-3}s^{-1}$ with 3.5 vol.% TiB whisker, and 439% obtained at 940℃、$5\times10^{-3}s^{-1}$ with 5 vol.% TiB whisker. The other results are shown in Fig. 5. The highest ductility obtained in 3.5vol.% TiB sheet, but average of ductility of 5.0 vol.% is higher than 3.5 vol.% at the same tensile conditions.

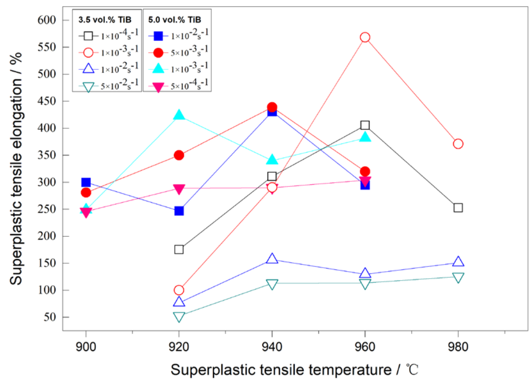

Fig.5 Effect of temperature and strain rate on ductility of composites sheet with 3.5 vol% and 5.0 vol.% TiB whisker

Fig.6 shows comparison of 3.5 vol% and 5.0% TiB whisker strain-stress curves as the same superplastic tensile parameters. Obviously, the flow stress of 3.5 vol% TiB whisker is lower than 5.0 vol % TiB whisker. However, the elongations with strain rate $1\times10^{-2}s^{-1}$ of 5.0 vol % TiB whisker have better elongations.

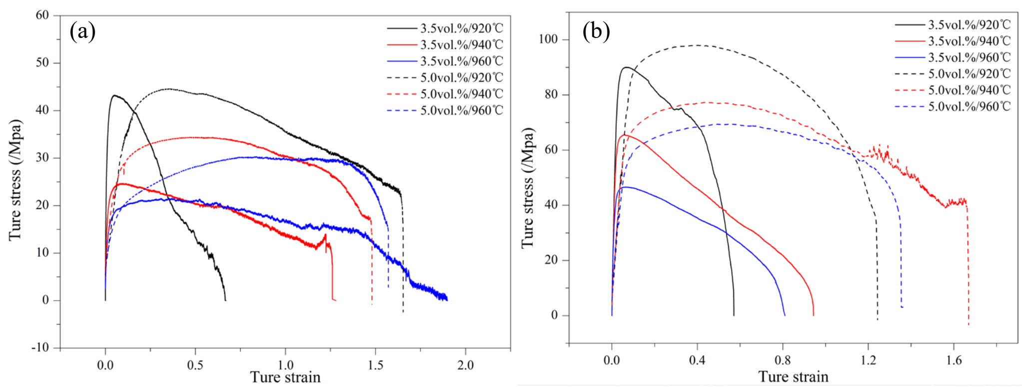

Fig.6 True stress-strain curves of the TiB_w/TA15 sheet at different temperature (a)$1\times10^{-3}s^{-1}$, (b)$1\times10^{-2}s^{-1}$

During superplastic tensile deformation, microstructure evolution of matrix alloy is deomained by α→β phase transformation and dynamic recrystallization, signigicant dynamic recrystallization occurred at second α lath phase, and sphered feature, which is almost the same as matrix alloy. Superplasticity of matrix will effect on the changes of TiB whiskers, the higher superplasticity of matrix, the better consistent deformation between whiskers and matrix, TiB whiskers are difficult to fracture during deformation. Much more debonding defects will occur at interficail between whiskers and matrix when decreasing compatibility deformation.

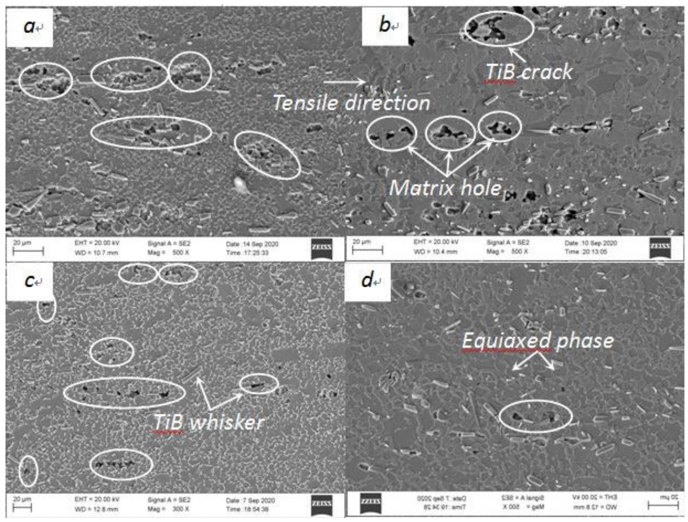

Fig.7 Superplastical microstructures deformed at different conditions
(a)900 ℃/5×10^{-3}s^{-1}, (b)960 ℃/5×10^{-3}s^{-1}, (c) 920 ℃/1×10^{-2}s^{-1}, (d) 920 ℃/5×10^{-4}s^{-1}

Significant effect of deformation strain rate on micro holes at higher strain rates, due to the worse consistent deformation at higher strain rate, dislocations will be stopped at whiskers, micro voids are easy to occur at here. Dynamic recreystillation not only could release the stress concentration, but also could close-up micro voids.

2.4 Diffusion bonding property and mechanism
TiB could increase the β transus with 30~40°C(1040°C), so diffusion bonding temperature of TMC also will increase accordingly. The optimized diffusion bonding temperature of matrix alloy is 920°C, and 1.0MPa gas pressure with 2 hours. Result of diffusion bonding rate examined by OM as shown in table1, the 95% diffusion bonding rate could be obtain by 950°C+2.5MPa and 960°C+1.6MPa with 3h.

Table 1 Diffusion bonding rate result with different parameters

No.	Temprature	Gas pressure	Time	Suface condition	Diffusion bonding rate
0	920°C	1.0MPa	2h	normal	>95%
1	950℃	1.6MPa	3h	normal	unbonded
2	950℃	1.6MPa	3h	grinding	30%
3	960℃	1.6MPa	3h	normal	60%
4	950℃	2.5MPa	3h	grinding	>95%
5	960℃	1.6MPa	3h	grinding	>95%

Fig. 8 shows the typical diffusion bonding microstructures with different rate. In fig.1(a), fine equiaxed grain with 100% diffusion bonding rate could be observed without TiB phase. Compared with as-received microstructure of TMC sheet, grains of matrix all turn to be more equiaxed with increasing of gas pressure and temperature, because of furnace cooling. Fairly uniform distribution of TiB whiskers with length less than 10μm. Interface with or without TiB whisker, the diffusion bonding mechanism the same as matrix if without TiB whisker, and the complete interface formed with TiB whisker due to the TiB whisker could continue react with matrix at phase boundary above 900°C. As result, we could expect all type of TiB whisker reinforced α or α+β type titanium matrix composites could be diffusion bonded. Surface roughness is a very critical effecting factor from experimental results; lower roughness could decrease parameters for diffusion bonding, because of TiB whisker existing like as a "needle", which will block the first stage of physic contact at high temperature.

As we know, fine equiaxed grain is the best microstructure for SPF and DB process, because of the theory of grain boundary sliding for superplasticity and density of grain boundary for diffusion bonding. However, present results revealed another microstructure also could have a good quality of bonding interface, the mechanism maybe more about phase boundary for diffusion of atom. Further research will prove the actual microstructure at diffusion bonding temperature through fast cooling to keep microstructure at high temperature. However, we have already investigated the extent of equiaxed grains depend on the pressure, which means that the outer pressure is a driven factor for transforming from α lath to equiaxed grains.

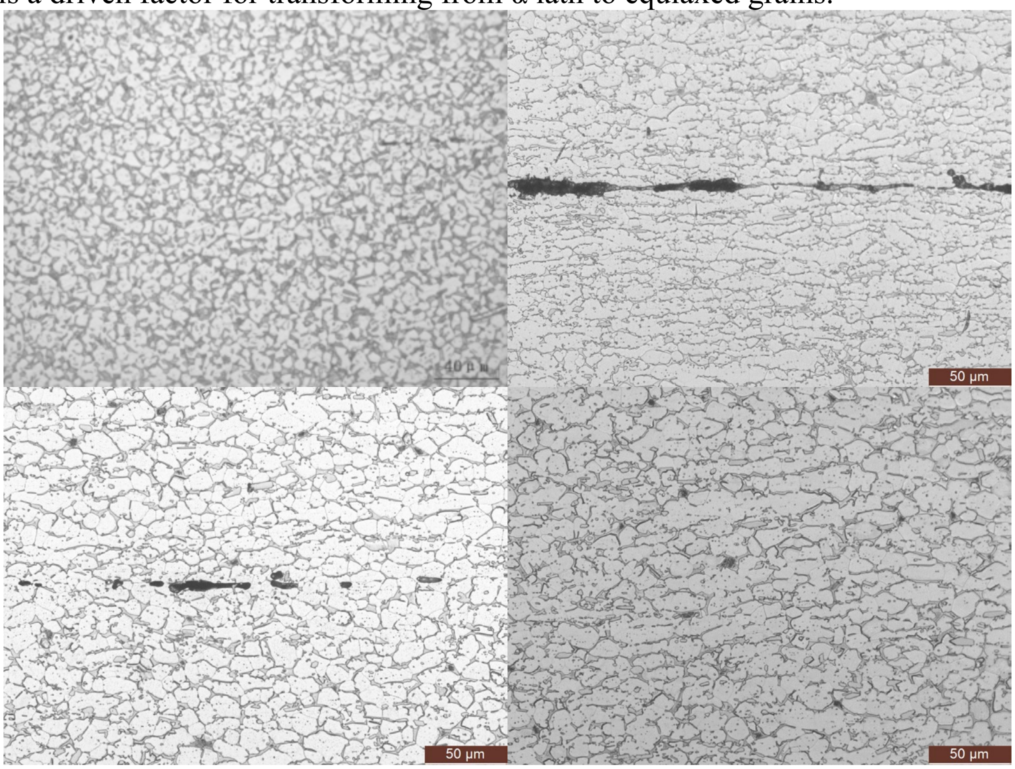

Fig. 8 Mcirostructure of diffusion bonding at (a) matrix alloy with 920℃+1.0MPa+2h; (b) TMC with 950℃+1.6MPa+3h; (c) TMC with normal surface at 960℃+1.6MPa+3h (d) TMC with grinding surface at 950℃+2.5MPa+3h

2.5 Four layers structure formed with SPF/DB

According the superplastic and diffusion bonding test results, parameters with 950~970℃, gas pressure with 1.6~2.0MPa, forming or diffusion bonding time with 2~3h. All other processing is same as traditional titanium alloys like as Ti64. The component with multiple ribs was successfully formed as showed in Fig. 9. Diffusion bonding interface between inner and outer sheets and ribs shows in Fig.3. Very good diffusion bonding quality could be observed, also finite decrease of thickness of inner sheet and without any maro holes formed in biggest deformation area of ribs.

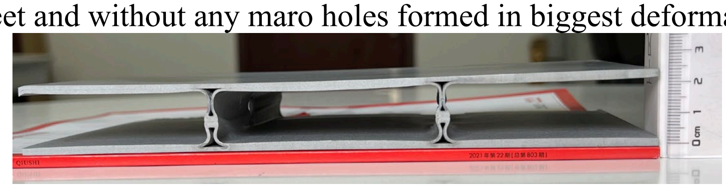

Fig. 9 SPF/DB formed component of in situ TMC

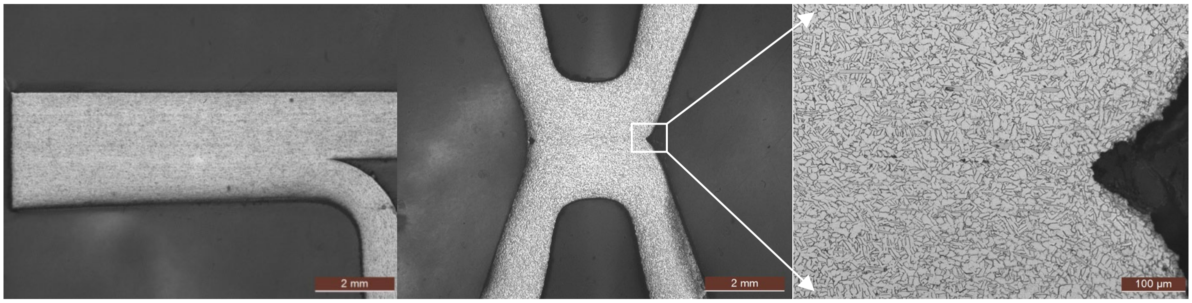

Fig.10 Macrostructure of diffusion bonding at surface and rib

4. Summary

(1) TiB whisker reinforced TMC is successfully fabricated with 1000mm wideness and 1.2mm thickness. Rolling processing is the critical factor to effect on mechnical properties. Rolling at above 100℃ from beta transformation temperature have a good tensile strength at RT and 650℃, but the ductility is related lower at RT.

(2) Obtained the best superplastic elongation with 569% and optimized diffusion bonding parameters with 95% rate, the combined process is used to successfully form multiple ribs component.

(3) Mechanism of diffusion bonding of in situ TMC depends on the phase boundary transformation and pressure.

(4) Evolution of matrix microstructure same as without TiB whisker, however, due to deformation compatibility between TiB whisker and matrix, after an extent deformation, the micro voids will be formed at the interface of matrix and whisker.

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