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Development of a Low-Cost Process for Manufacturing of Ti-Metal Matrix Composite by Roll-Diffusion Bonding

C. Testani and F. Ferraro

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Composite materials with titanium-alloy matrix are currently the class of material with the highest specific resistance at temperatures up to 800 °C. The main hurdle to their application is their final cost. Even if it is clear that the costs of constituent materials are decreasing due to volume production effects, the production processing costs remain high due to the batch production approach. Centro Sviluppo Materiali's (CSM) efforts have focused on the manufacturing process in order to obtain an innovative solution to reduce the manufacturing costs with respect to the hot isostatic pressing (HIP) process that represents the standard production process for this class of materials. The new approach can allow a cost reduction of about 40%; this result was obtained by developing an experimental "diffusion bonding" plant for co-rolling at high temperature in a superplastic rolling regime, sheets of titanium alloy and monofilament silicon carbide fabrics. The experimental pilot plant was proposed for patent with RM2006A000261 in May 2006. This paper describes the manufacturing phases and process results. Moreover, has been shown that the diffusion in the solid state was obtained in a process window that was at least 100 times faster than that of HIP. High-temperature tensile tests were carried out on specimens machined from metallic matrix composite materials produced with the roll-diffusion bonding (RDB) process. The samples produced were also submitted to electrochemical dissolution tests of the metallic matrix in order to verify the geometric integrity of the fibers inside the matrix after the bonding phase. The results achieved as well as the process knowledge acquired with the CSM pilot plant are the base for further development of industrial application of the titanium roll-diffusion bonding.

Keywords hot isostatic pressing, metal matrix composites, roll-diffusion bonding, titanium metal matrix composites

1. Introduction

Work with roll-diffusion bonding started in the United States in the early 1970s and was mainly focused on aluminum composites and on the manufacture of titanium composites using the pack rolling technique (Ref 1). This paper deals with innovative pilot-and-laboratory equipment, based on the roll-diffusion bonding process for the manufacture of titanium metal matrix composites (Ti-MMC) (Fig. 1). The manufacturing process is described as well as the development phases and product characterization. The innovative laboratory equipment has been patented (RM2006A000261 on May 2006) (Ref 2). The process itself consists of the manufacture of Ti-MMC samples monodirectionally reinforced with SiC fibers using a semicontinuous/continuous approach. The starting materials are titanium alloy sheet: Ti6Al4V and SP700 alloy and ceramic SiC continuous fibers. The materials are assembled as a lay-up of two metallic sheets with SiC fabric, as shown in Fig. 2.

During the roll-diffusion bonding phase, the just-assembled sample (precursor) has been put under a backward-and-forward strip tension under inert gas. The pressure, the rolling speed, and the mill-stand temperature are computer controlled to improve the bonding reaction, and this is the reason the process is called roll-diffusion bonding (RDB). The metallic strip plays the role of the composite matrix, and under the combined effects of temperature and pressure flows from the surfaces through the fiber interspaces inside the fabric, resulting in a complete metallic and ceramic-to-metal bonding.

The process phase is summarized as:

1. Heating up of a portion of the lay-up in the rolling-inert gas chamber
2. Plastic deformation of the metal matrix that starts the flow between the fibers
3. First chemical reactions (interdiffusion) at the fiber-matrix interfaces
4. Facing and contact between the two metal flows in the spaces between the fibers
5. Matrix recrystallization in the joining surfaces

The bonding process is stabilized and is completed in about 10 s. A further diffusion bonding phase is improved using post-heat-treatment that controls just the Ti-MMC strip temperature to improve the diffusion strength.

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C. Testani and **F. Ferraro**, Centro Sviluppo Materiali SpA, Via di Castel Romano 100, 00128 Rome, Italy. Contact e-mail: c.testani@c-s-m.it.

2. Pilot Equipment Design and Realization

The process window parameters needed for the design of the pilot equipment (rolling speed, strain rate, temperature, pressure, etc.) have been defined by literature data and experimental hot-compression tests including some pack-rolling tests. The design parameters adopted for the first dimensioning of the pilot equipment are reported in Table 1.

The large difference between the thermal capacities of the precursor and the rolling tools necessitates maintenance and control of the isothermal state in the contact arc (Fig. 1). A large effort was devoted to developing a technical solution to this problem, and a finite element method (FEM) simulation was used to design a set of possible solutions, make an inner comparison, and select the best solution for the superplastic flow and bonding in the rolling contact arc. In summary, from the plant design viewpoint, it was necessary to manufacture an inexpensive dynamic system capable of reaching a controlled temperature of 900 °C, in a homogeneous volume region, in inert gas, and at a pressure of at least 500 MPa in the rolling arc contact. The best solution consists of heating up a sealed chamber with inside the work rolls using electrical resistance; all other devices—microwaves, induction, and joule effect—proved to have higher complexity and costs. Thus it was possible to design a compact working region (electric resistance is distributed on a crown of about 240 spatial degrees on each work roll) with controlled Ti-MMC strip and rolling tool temperature.

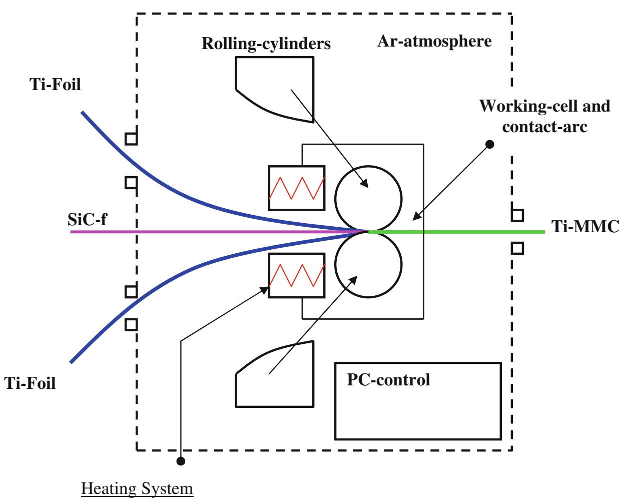
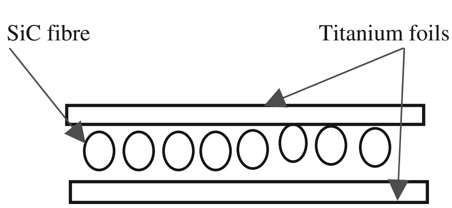


Fig. 1 Roll-diffusion bonding schematic



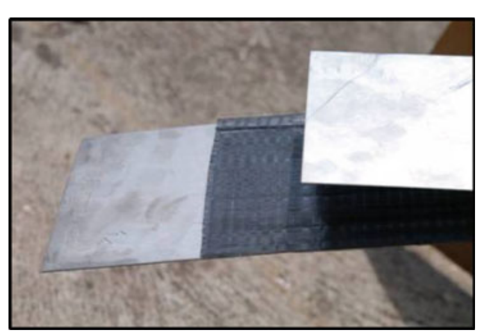


Fig. 2 Composite lay-up and preparation of a precursor for Ti-MMC

In particular, the large thermal mass of the rolling tools was able to heat up the Ti-MMC precursor, reaching 900 °C in about few tenths of seconds. To guarantee a safe process temperature of the work rolls, a cooling circuit was needed to prevent an excessive cooling effect, and it was necessary to design concentric work rolls: an inner force roll and external high-temperature-resistant alloy work rings, (Fig. 3). This solution offered two advantages: the heating of only the external special alloy rings and the manufacture of inexpensive standard tool steel inner-cooled work rolls. The space between inner and external tool was insulated with special shields to reduce thermal dispersions.

The geometric solution was the subject of a two-step thermal FEM validation (preliminary and detailed analysis), including also the computation of the rotation effect of the inner work roll on the external ring (Fig. 4). The MSC MARC code has been used for the thermal simulation with the rolling tools (work roll and ring) rotation. In this case, the simulation also took into account the radiation view factors between rolling tools and titanium sheet.

Table 1 Experimental reference parameters adopted for the pilot plant design

Exercise temperature, °C	Mean pressure in the contact arc, MPa	Time, s
900	500	6

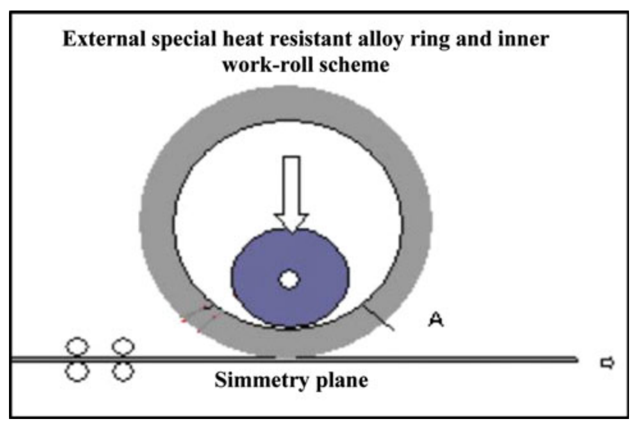


Fig. 3 Rolling cylinders with the splitting of the thermal tool (external ring) and the pressure tool (inner cylinder)

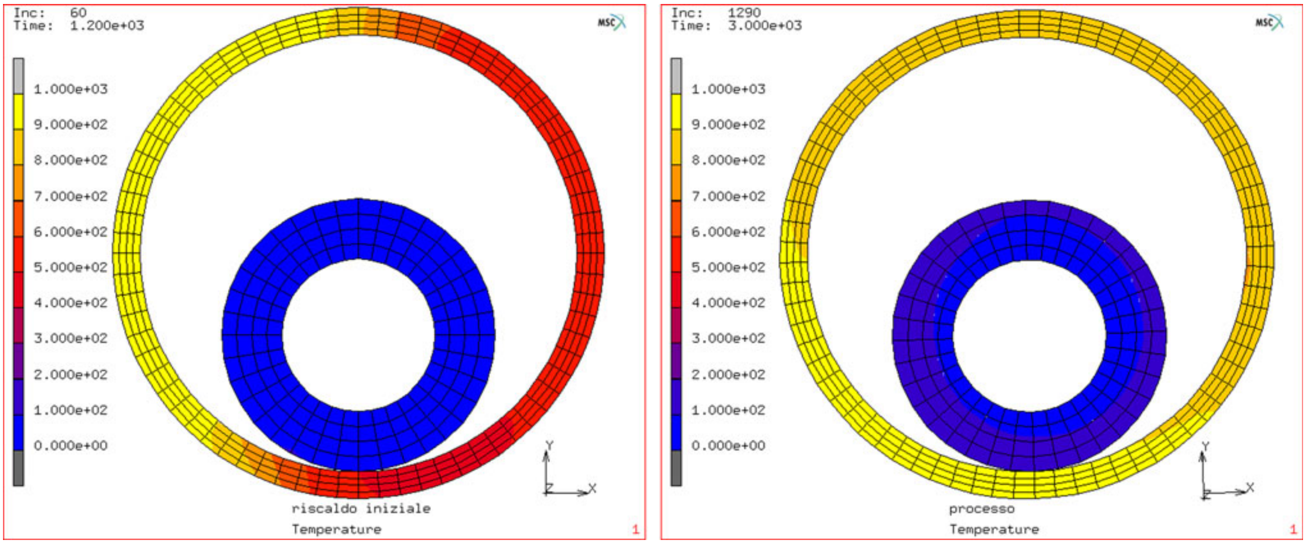


Fig. 4 Simulation of the thermal trend in degree Celsius during the counter-clockwise rotation

Fig. 5 Roll-diffusion bonding pilot plant (on the left) and detail of the mill stand

The work-roll rotation starts when the external ring high-temperature surface reaches a temperature that is sufficient to warrant at least 900 °C in the contact area. Centro Sviluppo Materiali S.p.A (CSM) developed the pilot/laboratory plant show in Fig. 5, on the base of the thermal simulation results. The core of the plant is the roll-diffusion bonding (RDB) chamber with a maximum separating load of 300 kN. The chamber is shown in Fig. 5, and when closed it works under 1.1 bar in inert gas (Ar) to avoid any contamination of the titanium sheets during the roll-bonding phase. The maximum operating temperature is 1150 °C in inert gas. An oxygen spectrometer continuously controls the quality of the atmosphere inside the chamber.

Another furnace (in inert atmosphere) is placed just after the mill stand to maintain the Ti-MMC sample at high temperature to improve the diffusion reaction and bonding.

The experimental tests conditions are summarized in Fig. 6, which also shows for comparison purposes the literature data available (Ref 3-5). The CSM plant produces roll-diffusion bonded samples with arc-contact times that are about 1000 s shorter than the hot isostatic press tests and/or hot pressing processes referred to in the literature data. In any case, it must be outlined that in the present plant configuration the effective process window for RDB requires a fine process control.

The roll-bonding optimal process temperature is about 30 °C lower than the temperature used in the hot isostatic pressing (HIP) process. This can be explained by taking into account that the diffusion at the Ti-matrix/SiC-fiber interface improves with a driving force caused by the rapid opening of clean surfaces during the plastic deformation and bonding, a phenomenon already known in literature (Ref 6).

3. Metallographic Analysis

The Ti-MMC samples were analyzed using optical (OM) and scanning electron microscopes (SEM). Figure 7 shows some images of roll-bonded samples where it is possible to see the full bond and complete interface recrystallization of the metal flow between the fibers.

The SEM analysis on the fiber/matrix interface was carried out to describe the diffusion thickness that is less than 1.5 μm

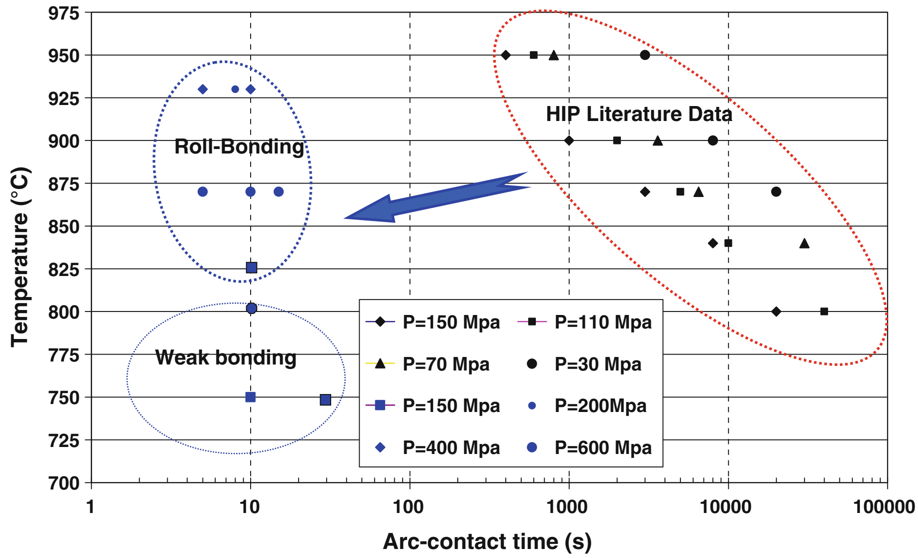


Fig. 6 Processing parameters

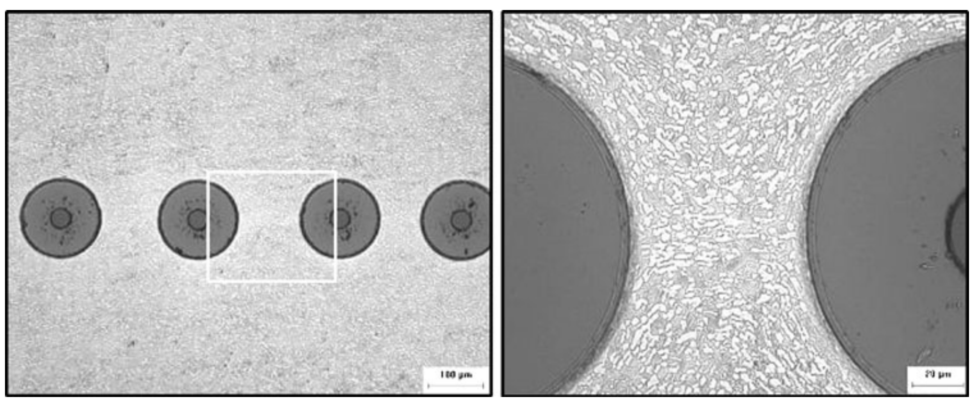


Fig. 7 Micrography of a SP700 alloy and SiC fibers. Right: a detail of the region where the bonding occurred

(Fig. 8). A wealth of literature reports a detailed discussion of the C diffusion into the matrix and a titanium diffusion to form a Ti_xSi_y chemical compound (Ref 7-11).

4. Fiber Analysis after Roll Bonding

One of the major concerns about the roll-bonding process is the long fiber integrity after the roll-bonding phase because of the high stresses reached in the contact arc region. To verify that the Ti-MMC fibers are not affected by macrocracking, it was decided to carry out chemical dissolution of the titanium matrix. The results obtained show that the resulting fibers were not cracked and that the metallic wire, used to weave the SiC fabric, takes part in the deformation process. The metallic wire in this case is a titanium-based alloy. Figure 9 shows a metallographic image in which the metallic wrap is plastically deformed on the fiber interface without any cracking.

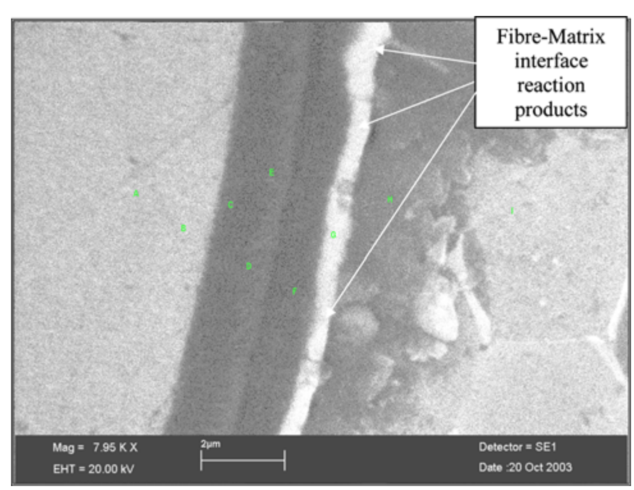


Fig. 8 Example of titanium-SiC surface interface

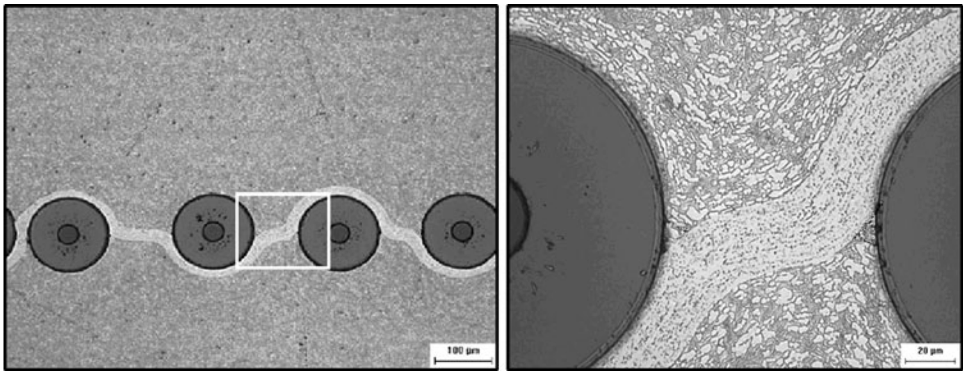


Fig. 9 Transversal section with the titanium warp-wire well deformed (view and a detail of the wire crossing two successive fibers)

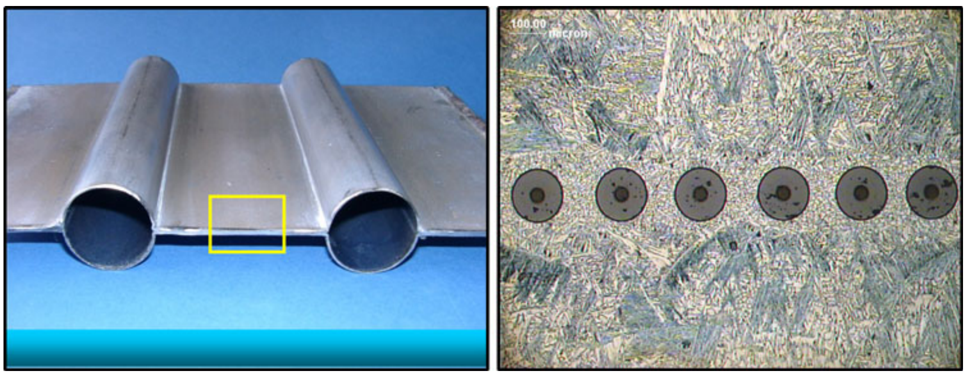


Fig. 10 Technological demonstrator with TI-MMC reinforcement

Table 2 Tensile tests results on specimens obtained with three manufacturing processes

Manufacturing process (fiber fraction: 10.6%)	Temperature, °C	Yield strength, MPa	Ultimate tensile strength, MPa
Foil-fiber-foil process HIP-DB. Process F-F-F	Ta	893 ± 10	1298 ± 20
	400	710 ± 12	876 ± 35
	600	543 ± 13	659 ± 26
	800	285 ± 21	329 ± 24
Thermal spraying and HIP-DB. Process T-S	Ta	853 ± 12	1152 ± 15
	400	728 ± 16	884 ± 23
	600	533 ± 14	589 ± 16
	800	276 ± 15	320 ± 15
Roll-diffusion bonding. Process RDB	Ta	898 ± 35	1282 ± 11
	400	712 ± 34	942 ± 27
	600	553 ± 27	596 ± 23
	800	315 ± 29	337 ± 36

Table 3 Main manufacturing costs for Ti-MMC

Main cost (1 m^2 of Ti-MMC)	Plasma spray + HIP	Fiber-foil lay-up + HIP	Pack rolling	Roll-diffusion bonding
Material	2570	6370	6370	6370
Thermal spraying	2600	0	0	0
Die (complete cycle)	1500	1500	1500	
HIP cycle cost	1600	1600	0	0
Rolling	0	0	1020	1250
Cost	9.170	9.470	7.870	6.370
RDB-savings	30%	49%	24%	1

5. Mechanical Characterization

Tensile specimens machined from the roll-diffusion bonded strips were mechanically characterized in tensile tests at room temperature up to 800 °C in accordance with ASTM Standard E 21 (Table 2).

For comparison, different specimens were prepared using two alternative processes: conventional hot isostatic pressing (HIP-DB) of a foil-fiber-foil lay-up (process F-F-F) and thermal spraying of SiC fabric with titanium alloy matrix followed by HIP-DB (process T-S). Thus it was possible to produce similar Ti-MMC tensile specimens with three different processes: same matrix, same alloy (SP-700); same fiber volume fraction (about 10% vol); and same fiber mono-orientation (0° fibers). Table 2 reports the mean values on three tests for each methodology. The values recorded are comparable and also close to those reported in literature for the same fiber-volume fraction (Ref 3). The Ti-MMC material obtained by RDB is ready to be used as reinforcement in any superplastic formed (SPF-DB) component with the approach to insert the aforementioned Ti-MMC in the higher-stressed region. Several SPF-DB tests were carried out using the RDB strip, and it was possible to obtain a SPF "technological demonstrator," as shown in Fig. 10 with a metallographic detail of the reinforcement with Ti-MMC. The SPF parameters are those for a standard production without any adjustment.

The last activity to validate the RDB process was the cost analysis comparison for the three manufacturing processes. The cost obtained for RDB was normalized to 1. Our assessment shows that it is possible to reduce down to the 40% the production costs with respect to the F-F-F process (Table 3). Obviously, a large contribution to the total costs is the starting material costs and ‘‘constrained availability’’ that could be reduced in the future.

Another main reason for these results is clearly the die sealing and opening operations that represent the core of F-F-F process and that are unnecessary for RDB.

6. Conclusions

The laboratory pilot plant realized at CSM shows that it is possible to manufacture titanium-based composites with the roll-diffusion bonding process. The process consists of cobonding of two titanium alloy strips with a SiC (mono-oriented) fabric under strictly controlled parameters in terms of temperature/pressure and strain rate. The properties of the Ti-MMC are shown to be comparable with those obtained using well-known processes such as fiber-foil and hot isostatic pressing. The assessment of cost reduction has shown that a 40% saving is possible, and this fact could be a real chance for this class of expensive materials to be used in the next generation of titanium airplanes. A very simple technological demonstrator has been manufactured using SPF-DB process with a Ti-MMC insert to show the great opportunity for the Ti-MMC and in particular for RDB Ti-MMC.

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