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Characterization of Ni_3Al Alloy Fabricated by Thermal Explosion/Hot Extrusion Technique

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In the present paper, the trace B doped Ni_3Al alloy was prepared by the thermal explosion and hot extrusion (TH/HE) technique. Its microstructure characterization and mechanical properties were carried out by OM, XRD, TEM, compression and microhardness tests. Microstructure examinations exhibit that the TH/HE technology has transformed the powders into Ni_3Al alloy with less porosity. In the synthesized part, coarse and ultrafine Ni_3Al grains constitute the dual-scale grain structure with segregated Al_2O_3 particles along grain boundary. TEM observations reveal that γ-Ni, Al_2O_3 and Ni_3Al phases coexist in the synthesized part. Moreover, the Al_2O_3 particles in the synthesized part have α-Al_2O_3 and γ-Al_2O_3 two crystal structures. In addition, there is a transition region along the interface of Al_2O_3 and Ni_3Al grain. The subsequent hot extrusion optimizes the microstructure of the extruded part by homogenizing the Ni_3Al grain, uniformly distributing Al_2O_3 and generating massive substructures. The great deformation results in the pile-up of dislocations, intersected dislocations and substructures. Moreover, the movement of dislocation promotes the formation of micro-twinning in the Ni_3Al grain, which is accompanied with the stacking faults and nano-structure. The mechanical properties exhibit that the microstructure optimization caused by the TE/HE technique improves the compressive ductility and strength of the Ni_3Al alloy obviously.

Keywords: Ni_3Al, thermal explosion, hot extrusion, microstructure, mechanical properties.

1. INTRODUCTION*

Intermetallic compounds are the kind of materials which possess the special properties in some aspects, so they have been paid much attention and many researches have been carried out [1 – 4]. As a kind of intermetallic compound, Ni_3Al attached so much attentation due to its excellent properties at high temperature, such as high melting point, good creep resistance, excellent corrosion and oxidation resistance [5 – 7]. Therefore, it has been considered as a promising structure material to use in aircraft engine [8]. Despite of these attractive advantages, the brittle fracture, processing problem and low low ductility of the Ni_3Al are the major difficulties that handicap its wide application [9, 10]. In order to solve these key problems, many methods have been applied and many kinds of Ni_3Al based alloys [11 – 13].

The previous research of Liu exhibited that the trace addition of B could improve the ductility of the Ni_3Al greatly with 24 % (at.%) Al [14]. While the research on the Ni_3Al revealed that the moisture environment resulted the embrittlement of the Ni_3Al [15]. Therefore, the addition of B would resist the influence of moisture on the grain boundary of Ni_3Al and improve its ductility. Though the B addition in Ni_3Al could increase its ductility, however its yield strength still not high enough. According to the recent investigations [16 – 18], the microstructure refinement was a convenient way to increase the strength without detrimental to the ductility. Previously, the study on the Ni_3Al alloy exhibited its mechanical properties could be improved further by the grain refinement and dispersoid particles [7, 19]. Among the material processing technologies, the powder metallurgy is recognized as an attractive method to fabricate the Ni_3Al alloy with strengthening particles, because of its advantages of microstructure refinement and near-net forming [20, 21]. The research of Yeh exhibited that the Ni_3Al alloy could be fabricated by self-propagating synthesis [22]. While the A. Antolak-Dudka et al. [23] fabricated the bulk nanocrystalline Ni_3Al alloys by hot-pressing consolidation of mechanically alloyed powders. However, the researches of Sheng [12, 18, 24] revealed that there was porosity in the Ni_3Al or NiAl based alloy prepared by powder metallurgy. In general, the fabricated specimen by powder metallurgy would be treated with hot isostatic pressing (HIP), but it would result in the coarsening of microstructure. The recent studies [12, 20] demonstrated that the hot extrusion following the synthesis could densify the specimen and decrease the porosity obviously. In addition, based on the former research [14], the addition of 0.2 – 1 % (at.%) boron should be appropriate selection, which had the optimal improvement on the ductility. Therefore, in the present research, the thermal explosion and hot extrusion (TE/HE) is employed to fabricate the Ni_3Al alloy with 0.3 % (at.%) boron addition. Its microstructure is characterized by the transmission electron microscopy and its mechanical properties are analyzed as well.

2. EXPERIMENTAL DETAILS

The elemental nickel powder (with an average size of 0.9 μm), aluminum powder (1.5 μm) and boron powder

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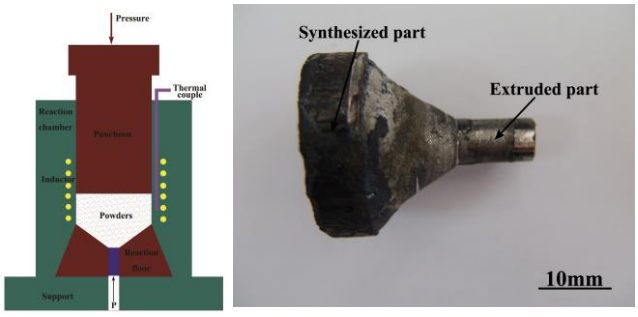
(3 μm) were used as the initial materials. These powders (Ni:Al:B = 74.7:25:0.3, atomic ratio) were dry mixed by the ball milling for three hours. The mixed powders were put into the TE/HE synthesis system, as shown in Fig. 1 a. The powders were compacted by the puncheon to get a relative densified sample. Then the densified sample was heated by induction coil rapidly to 760 K to ignite the reaction synthesis. During the heating and synthesis, the thermocouple in the TE/HE synthesis system was used to detect the temperature of the compacted powders. When the detected temperature was higher than 2000 K, it indicated the reaction synthesis had begun. Then, a force of 400 MPa would be exerted on the reaction puncheon to extrude the synthesized Ni_3Al out of the reaction floor through a hole with the diameter of 6 mm. During the extrusion, a back force was exerted on the extruded part to ensure its compactness. The as fabricated sample is shown in Fig. 1 b.



a b

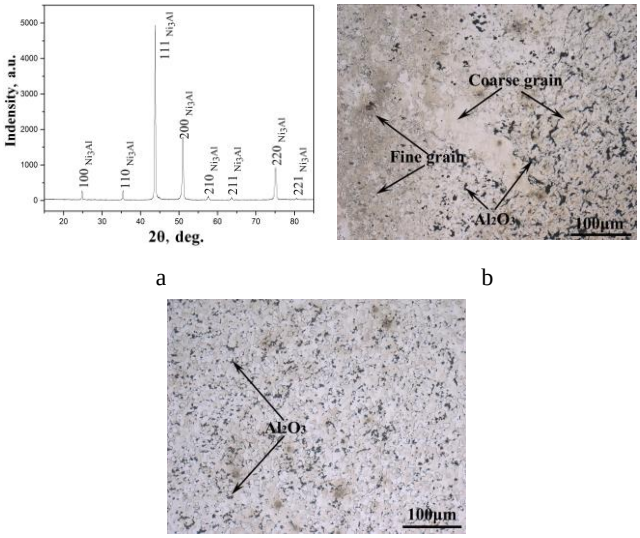
Fig. 1. a – schematic diagram of the thermal explosion and hot extrusion synthesis system; b – appearance of the synthesized sample

Specimens for microstructure characterization and mechanical properties tests were cut from the synthesized part and extruded part. The OLYMPUS GX41 optical microscope (OM) was used to perform microstructure characterization. The specimen for OM observation was prepared by mechanical polishing and chemical etching with the acidic mixture ($CH_3COOH/HNO_3/HCl$=8:4:1). The resultant phases in the Ni_3Al alloy were analyzed by X-ray diffraction (XRD) with a Cu radiation at 40 kV and 40 mA. JEOL-2100 high-resolution transmission electron microscope (TEM) was employed to carry out the TEM characterization and the specimen was cut from alloy with the thickness of 0.4 mm. Then the specimen was polished to 30 μm and shaped into φ3 mm in size followed by twin-jet electropolishing. Due to the small size of the synthesized sample, compression test and microhardness were adopted to evaluate its mechanical properties. Compression sample (4 mm × 4 mm × 6 mm) was cut from the synthesized sample and mechanically grounded by 800-grit SiC abrasive. Gleeble 1500 was employed to carry out the compression test at the strain rate of 1×10^{-3}/s. Hardness evaluation was performed on the HV-1000 Vicker microhardness tester with dwell time of 20 s and load of 150 g.

3. RESULTS AND DISCUSSION

3.1. Microstructure characteristics

The Ni_3Al alloy with trace B addition was successfully fabricated by TE/HE technique. The X-ray diffraction pattern of the Ni_3Al alloy is analyzed based on the powder diffraction data of Ni_3Al structure (JCPDS 65-0144) [25], as shown in Fig. 2 a. Clearly, the elemental powders have fully reacted and been transformed into Ni_3Al phase by the TE/HE process. Moreover, it can find that the Ni_3Al prefers to grow along the (111) crystal plane. Except the Ni_3Al phase, just a little peak of Al_2O_3 phase can be found in the X-ray diffraction pattern. OM image of the synthesized part and the extruded part of the TE/HE synthesized Ni_3Al alloy are shown in Fig. 2 b and c, respectively.



a b

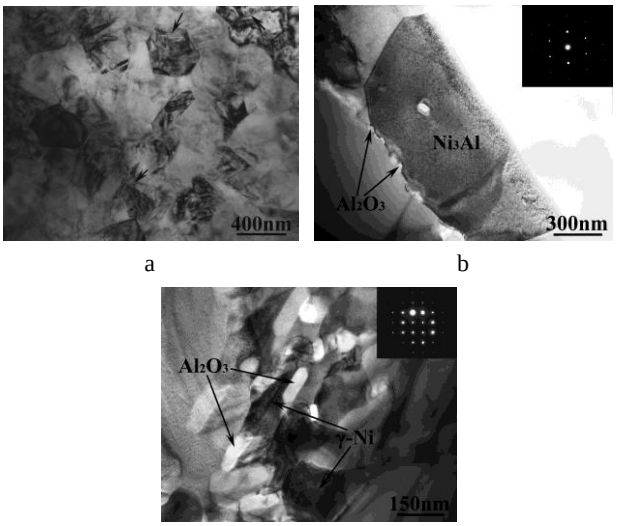
c

Fig. 2. a – X-ray diffraction patterns of Ni_3Al alloy prepared by TE/HE; b – OM image of the synthesized part; c – OM image of the extruded part

Both parts of the synthesized alloy are well compacted with low porosity, but the extruded part has more homogeneous microstructure than the synthesized part. In the synthesized part, there are multiscale grains with grain size from 10 μm to 100 μm. In the marginal region of the synthesized part, the ultrafine grain less than 5 μm is the main feature, which may be ascribed to the high cooling rate resulted by the mould of TE/HE synthesis system. With the region approaching to the centre of synthesized part, the microstructure becomes coarse and dual-scale grains appear. The coexistence of coarse and fine grains should be attributed to the high reaction temperature and low heat transfer rate, because the marginal region restrains the heat transfers rapidly. The residual heat promotes the growth of the Ni_3Al grain. Due to the competition of grain growth, some ones grow rapidly and become the coarse grain. In the region far from the margin, the microstructure is mainly composed of dual-scale grain and Al_2O_3 particles. Moreover, it can be found that the Al_2O_3 particles mainly distribute on the grain boundaries, but some agglomerate obviously. In the extruded part, the grain is homogeneous with the average size of 10 μm. The

Al_2O_3 particle in the extruded part is fine and distributes uniformly. The gross statistics on the Al_2O_3 particles in the synthesized Ni_3Al alloy indicates its volume percent is about 4 %. The formation of Al_2O_3 particles should be ascribed to oxidation during the powders milling and thermal explosion process, because the TE/HE system have no protective atmosphere. The oxygen could infiltrate into the green compact during the initial compacting. Then the Al_2O_3 dispersoids would be generated due to its priority [26]. The presence of Al_2O_3 dispersoids is beneficial to the microstructure refinement by forming necklace like precipitates along grain or phase boundary [27, 28].

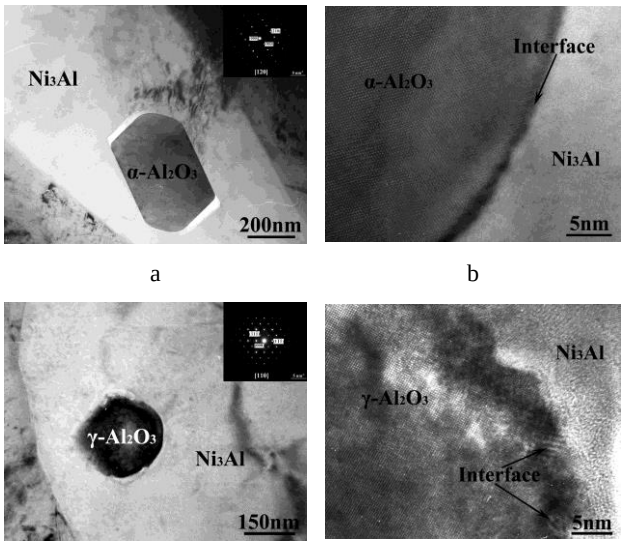
The results of TEM observations on the synthesized part of the Ni_3Al alloys are presented in Fig. 3. Different from the OM observation, TEM analysis exhibits that the matrix contains nanostructure, as shown in Fig. 3 a. The ultrafine Ni_3Al grain has the size of hundreds of nanometers. Such refined microstructure may be partly attributed to the deformation induced by the following hot extrusion that forces the synthesized part to deform and promotes the subgrains. The selected area diffraction pattern (SADP) confirms the formation of the Al_2O_3 dispersoids, as shown in Fig. 3 b. The Al_2O_3 dispersoids prefer to distribute along the grain boundary, which could contribute to the grain refinement. The aggregated Al_2O_3 dispersoids in the matrix can generate the barrier effect, which prevents the growth of Ni_3Al grain at high temperature. Moreover, they are also helpful to the fragmentation of Ni_3Al phase, due to its high strength at high temperature. They would result in the pinning effect and separate the bulk Ni_3Al phase during the hot extrusion. The observations on the Al_2O_3 dispersoids aggregated region reveals the formation of γ-Ni phase, as shown in Fig. 3 c. Except the segregation of Ni powders, such a phenomenon should be ascribed to the reaction of oxygen with aluminum during the synthesis process, which causes the deficient of Al.



a b c

Fig. 3. a – TEM image of ultrafine Ni_3Al grains in the synthesized part; b – morphology of Al_2O_3 particles along grain boundary (Inset pictures show the SADP of Al_2O_3); c – formation of γ-Ni phase around Al_2O_3 particles (Inset pictures show the SADP γ-Ni)

TEM observations on the Al_2O_3 particles exhibit they contain two kinds of crystal structure, as shown in Fig. 4. The SADP has confirmed that one is α-Al_2O_3 with the hexagonal crystal structure (a = b = 0.4758 nm, c = 1.299 nm) and R3c space group, as shown in Fig. 4 a. Based on the recent research [29], the α-Al_2O_3 is the most stable phase in all Al_2O_3 crystals. The high-resolution TEM (HRTEM) observation on the interface of α-Al_2O_3 and Ni_3Al reveals that there is a transition area about several nanometers, as shown in Fig. 4 b. In the transition area, the atoms array changes a little compared with that in the center of α-Al_2O_3. Combining SADP and TEM observations, the other Al_2O_3 is determined as the γ-Al_2O_3 with the face-centered crystal structure (a = b = c = 0.7948 nm) and Fd3m space group, as shown in Fig. 4 c. The γ-Al_2O_3 is a metastable phase that would transform into α-Al_2O_3 during heat treatment. HRTEM observation on the interface of γ-Al_2O_3 exhibits that it has a zigzag pattern and contains interface dislocations, as shown in Fig. 4 d. Additionally, along the interface some subgrains start to form. The changes of the interface of Al_2O_3 should be ascribed to the great lattice difference. It is the rapid synthesis process that results in the presence of the two kinds of Al_2O_3. During the synthesis, the high temperature and high pressure in the short time promote the formation of Al_2O_3 particles with the different structure, but the high cooling rate handicaps the phase transformation and reserve theses Al_2O_3 crystals.



a b c d

Fig. 4. a – bright-field TEM micrographs of the α- Al_2O_3 particle (Inset image showing the SADP of α-Al_2O_3 precipitate); b – HRTEM image of α-Al_2O_3/Ni_3Al interface; c – bright-field TEM micrographs of the γ- Al_2O_3 particle (Inset image showing the SADP of γ-Al_2O_3 precipitate); d – HRTEM image of γ-Al_2O_3/Ni_3Al interface

TEM observations on the extruded part of the TE/HE synthesized Ni_3Al alloy reveal that the Ni_3Al has experienced great deformation, as shown in Fig. 5 a. The hot extrusion process forces the Ni_3Al grain to extend along the extrusion direction, so some Ni_3Al phase exhibits long strip shape. Compared with the synthesized part, the grain size of the extruded part becomes finer, and the distribution of Al_2O_3 dispersoids becomes more

homogeneous. Then it can be deduced that the extrusion deformation and Al_2O_3 dispersoids work together results in the microstructure refinement. Due to the short time of TH/HE and great deformation, it is impossible for the alloy to have a recrystallization. Therefore, massive dislocations can be observed in the extruded part, as shown in Fig. 5 b. The dislocations intersect with each other along the grain boundary, which promotes the formation of the substructure. Obviously, the original grain boundary could effect as the barrier of the dislocations. It is difficult for the dislocations to traverse the grain boundary. In the bulk Ni_3Al grain, it still can find the dislocation arrays and their intersection, as shown in Fig. 5 c.

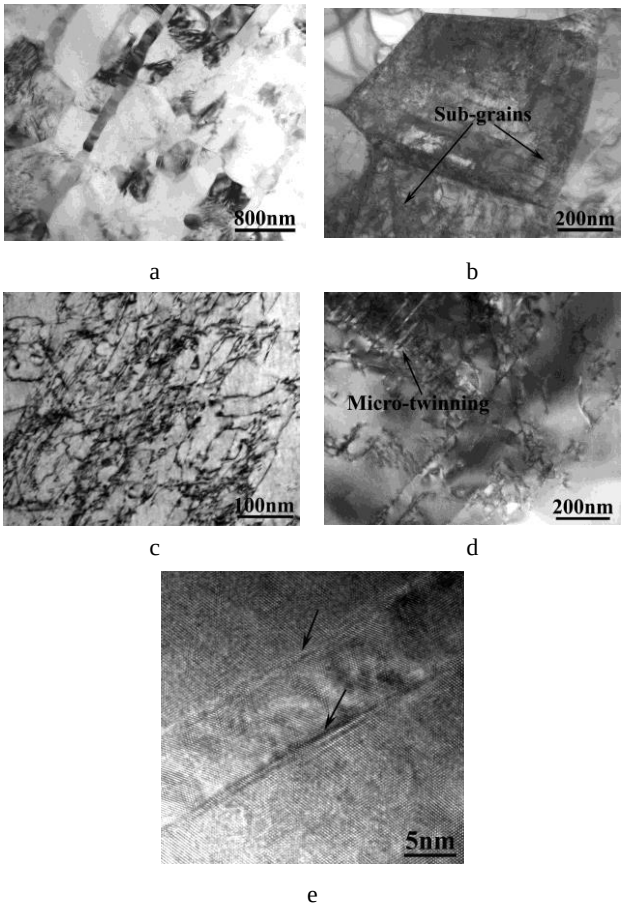


Fig. 5. a – TEM images of the extruded part showing the deformation of the Ni_3Al phase; b – irregular dislocations and subgrains along grain boundary; c – intersected dislocation arrays inside Ni_3Al grain; d – formation of microtwinning in Ni_3Al crystal promoted by dislocation; e – HRTEM of microtwinning boundary

Such intersected and tangled dislocations in the Ni_3Al grain should be attributed the great deformation during the extrusion process. Moreover, these dislocations are also beneficial to the strength and promote the crystal defects. As shown in Fig. 5 d, the micro-twinning is generating in the region with great pile-up dislocations and tangled dislocations. It is interesting that the twinning begins companied with the dislocations movement. HRTEM observation on the micro-twinning finds that stacking faults seems to form along the twinning boundary, as shown in Fig. 5 e. Near the micro-twinning region, it also can find that there are some substructures with several nanometers. It indicates that the great deformation promotes micro-twinning and substructure as well. In general, the microstructure evolution of Ni_3Al alloy during TE/HE should be ascribed to the great deformation, short synthesis time and high cooling rate. The short synthesis time and high cooling rate result in the diversified structure and phase. So many kinds of phases reverse in the synthesized part. The great deformation during hot extrusion compels the alloy deforms along the extrusion direction, which would promote the anisotropic characteristic. Then many crystal defects generate in the alloy.

3.2. Mechanical properties

The room-temperature mechanical properties and microhardness of the TE/HE synthesized Ni_3Al alloy are shown in Table 1. Moreover, the mechanical properties of Ni_3Al alloy fabricated by combustion synthesis in former research [30] are also listed in the Table 1. From the data, one can find that the TE/HE process improves the mechanical properties of the Ni_3Al alloy obviously, especially the strength. The improvement should be ascribed to the microstructures optimization by the TE/HE process. The fine Ni_3Al grain, trace B addition, Al_2O_3 dispersoids and massive crystal defects all contribute to the mechanical properties. According the previous researches [8, 14, 31, 32], the trace B addition in the Ni_3Al alloy with 24 % (at.%) Al increased the ductility obviously by improving the cohesion of grain boundary. In the present research, though trace B is added in the Ni_3Al alloy, however the milling and thermal explosion would preserve the B in the grain [12, 33, 34]. Therefore, the grain boundary strengthening by the B is restrained. Moreover, the Al content of present research is 25 % (at.%), which influences the ductility improvement further. Additionally, the presence of Al_2O_3 dispersoids also exerts negative influence on the compressive ductility. The widely distributed Al_2O_3 particle in the TE/HE synthesized Ni_3Al alloy contributes to the strength, but its non-uniform distribution, especially the segregated Al_2O_3 dispersoids, would lead to great stress concentration and decrease its compressive ductility. Then it can be understood that the compressive ductility of TE/HE Ni_3Al alloy is not improved significantly, compared with the Ni_3Al alloy without B addition. However, the comparison of the synthesized part and extruded part of the TE/HE prepared Ni_3Al alloy exhibits that the subsequent hot extrusion increases the yield strength a little but enhances the compressive strain obviously. Based on the microstructrue observation above, it can be find that the hot extrusion eliminates the dual-scale grain and homogenize the grain size. According to the former investigation [35 – 37], the dual-scale grains decrease the synchronization between grains during deformation, which may result in great dislocation accumulated in the coarse grain boundary and the crack initiation. The homogeneous grain in the part would be beneficial to the plastic deformation. In fact, the grain homogenization could be attributed to the presence of Al_2O_3 dispersoids partly. During the hot extrusion, the rheological behavior of Ni_3Al alloy compels the Ni_3Al grain to deform along the extrusion direction.

Table 1. Room-temperature mechanical properties and microhardness of the TE/HE synthesized Ni_3Al alloy and the combustion synthesized Ni_3Al alloys

Alloy	Yield strength, MPa	Compressive strength, MPa	Compressive strain, %	Microhardness, HV
Synthesized part	405	1350	23	280
Extruded part	450	1430	35	300
Ni_3Al [17]	230	680	15	220

The combination of grain torsion and pinning effect of Al_2O_3 dispersoids promote the fragmentation of bulk Ni_3Al grain in the neck of the mould. Moreover, the necklace distribution of Al_2O_3 dispersoids along grain boundary restrain the overgrowth of grain. In addition, the great amounts of dislocations and subgrains generated in the extruded part contribute much to the strength.

4. CONCLUSIONS

1. The trace B doped Ni_3Al alloy is prepared by the thermal explosion and hot extrusion synthesis technique. In the synthesized part, coarse and ultrafine Ni_3Al grains comprise the dual-scale grain structure with segregated Al_2O_3 particles along grain boundary. Moreover, the Al_2O_3 formed in the synthesized part contains α-Al_2O_3 and γ-Al_2O_3 two crystal structure, possessing a transition region along the interface.
2. The subsequent hot extrusion optimizes the microstructure of the extruded part by homogenizing the Ni_3Al grain, uniformly distributing Al_2O_3 and generating massive substructures. Moreover, the great deformation promotes dislocations movement and results in the formation of micro-twinning.
3. Compared with the combustion synthesized Ni_3Al alloy, the thermal explosion and hot extrusion synthesized Ni_3Al alloy owns better mechanical properties. Moreover, the extruded part possesses the highest compressive properties

Acknowledgments

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