

# MICROSTRUCTURE AND MECHANICAL PROPERTIES OF (Nb, Ti) C-NICKEL BASE SUPERALLOY<sup>①</sup>

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**ABSTRACT** (Nb, Ti) C-nickel base superalloy was prepared via hot-isostatic pressing (HIP). Its flexural strength was tested by means of three-point bending. The flexural strengths at room temperature and at 1000 °C are  $1318 \pm 158$  MPa and  $506 \pm 50$  MPa respectively. The sample was observed with transmission electron microscopy (TEM) and the mechanisms of reinforcements were discussed.

**Key words** mechanical properties microstructure titanium carbide

## 1 INTRODUCTION

TiC-Ni, as a typical kind of cermet, has been studied in detail<sup>[1-3]</sup>. However, as a high-temperature structural material, the fracture toughness of the TiC-Ni cermet is relatively low. One main reason for its low fracture toughness is that the compatibility and binding between TiC ceramic and Ni metal phase are very weak. During the past decades, many methods have been employed to improve the compatibility and binding between TiC and Ni, such as doping  $Mo_xC$  and  $Nb_xC$ <sup>[4]</sup> and using Ti(C, N) instead of TiC<sup>[5]</sup>. Bogomolov<sup>[6]</sup> studied Mo-doped TiC ceramic and found that the dopant Mo resulted in some improvement of brittleness of the TiC ceramic. We have used Nb as dopant. Because the melting temperatures of both Nb and TiC are very high, Nb is difficult to be doped into TiC via general methods. By using arc melting technique, we investigated (Nb, Ti) C-Ni (abbreviated as TN1) cermets<sup>[7]</sup>. Our compatibility test<sup>[8]</sup> showed that the compatibility between (Nb, Ti) C and Ni was better than that between  $TiC_{0.78}$  and Ni, and (Nb, Ti) C-Ni cermet exhibited better mechanical properties than  $TiC_{0.78}$ -Ni<sup>[7]</sup>. The backscattered electron (BSE) images also

showed that most of the (Nb, Ti) C grains in the cermet consist of two parts, i. e., black interior and gray surrounding layer, which is called as surrounding structure; and the electron microprobe analysis of the surrounding structure showed that the grain interior is rich in Ti and poor in Nb, while the surrounding layer is rich in Nb and poor in Ti. Discrete Variational  $X_\alpha$  calculation of this structure showed that the content of metallic bonds in the surrounding layer is higher than that in the grain interior<sup>[9]</sup>. Therefore, the surrounding layer improved the binding between TiC ceramic phase and Ni phase, which is beneficial to increasing the mechanical properties of the cermet.

Because the formation of TiC from Ti and C is a very strong and rapid exothermic reaction process, and the combustion temperature can be as high as 3000 K<sup>[10]</sup>, we also doped TiC with Nb by means of thermal explosion and investigated (Nb, Ti) C-35Ni (abbreviated as TN2) and (Nb, Ti) C-35Ni( $Y_2O_3$ ) (abbreviated as TNY) cermets<sup>[11]</sup>. Our results showed that in the (Nb, Ti) C ceramic grains in these two systems no surrounding structure can be seen to exist;  $Y_2O_3$  addition is of benefit to reducing the pore in the cermet and has positive effect on

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improving the mechanical properties of the cermet, especially on its flexural strength at high temperature. In this study, we used nickel base superalloy instead of Ni and investigated (Nb, Ti) C-nickel base superalloy (abbreviated as TNS) composite.

## 2 EXPERIMENTAL

(Nb, Ti) C powder was prepared by means of thermal explosion. Powdered mixtures of titanium, niobium and graphite (Ti: Nb: C= 0.936: 0.064: 0.73 (mole ratio)) were combusted in argon. The size of the product, (Nb, Ti) C, was about 2  $\mu$ m after ball-milling for 1 h. The composition of nickel base superalloy is listed in Table 1. The composition of TNS was 65% (in

**Table 1 Composition of nickel base superalloy( %, in mass)**

Ni	Cr	W	Mo	Ta	Al	Ti	Zr	B	T <sub>2</sub> O <sub>3</sub>
68.74	15	4	2	2	4.5	2.5	0.15	0.01	1.1

mass) (Nb, Ti) C and 35% nickel base superalloy. The powders were homogenized in alcohol for 10 h, and then dried under vacuum. Green bodies were produced by cold isostatic pressing (at 300 MPa). Specimens were fabricated by vacuum sintering (about 133.3 Pa) at 1430 °C for 60 min, and then hot isostatic pressing (HIP) under 152 MPa in argon at 1390 °C for 45 min. The resulting cylinders were cut into bars of 34 mm  $\times$  3 mm  $\times$  4 mm for flexural strength test and 24 mm  $\times$  2 mm  $\times$  4 mm with a preprepare surface groove measuring 0.15 mm wide by 2 mm deep for fracture toughness test. The flexural strength and fracture toughness of specimens were tested by three-point bending over 30 mm and 20 mm spans, with across head speed of 0.5 mm/min and 0.06 mm/min respectively. In the high temperature flexural test, the specimens were held at the set-up temperature for 30 min before loading to ensure thermal equilibrium. Hardness was determined by conventional method. The foils suitable for TEM observation were prepared from the specimen. Thin disks (about 300  $\mu$ m thick by 4 mm in diameter)

were mechanically polished. Ion-thinning was carried out on disks of 50 mm thick by 3 mm in diameter in a commercially available ion-thinner (600-CDIF model) using argon ions at 8 kV. Foils were examined in H-600 STEM/EDX-PV 9100 electron microscope at 100 kV.

## 3 RESULTS

### 3.1 Mechanical properties

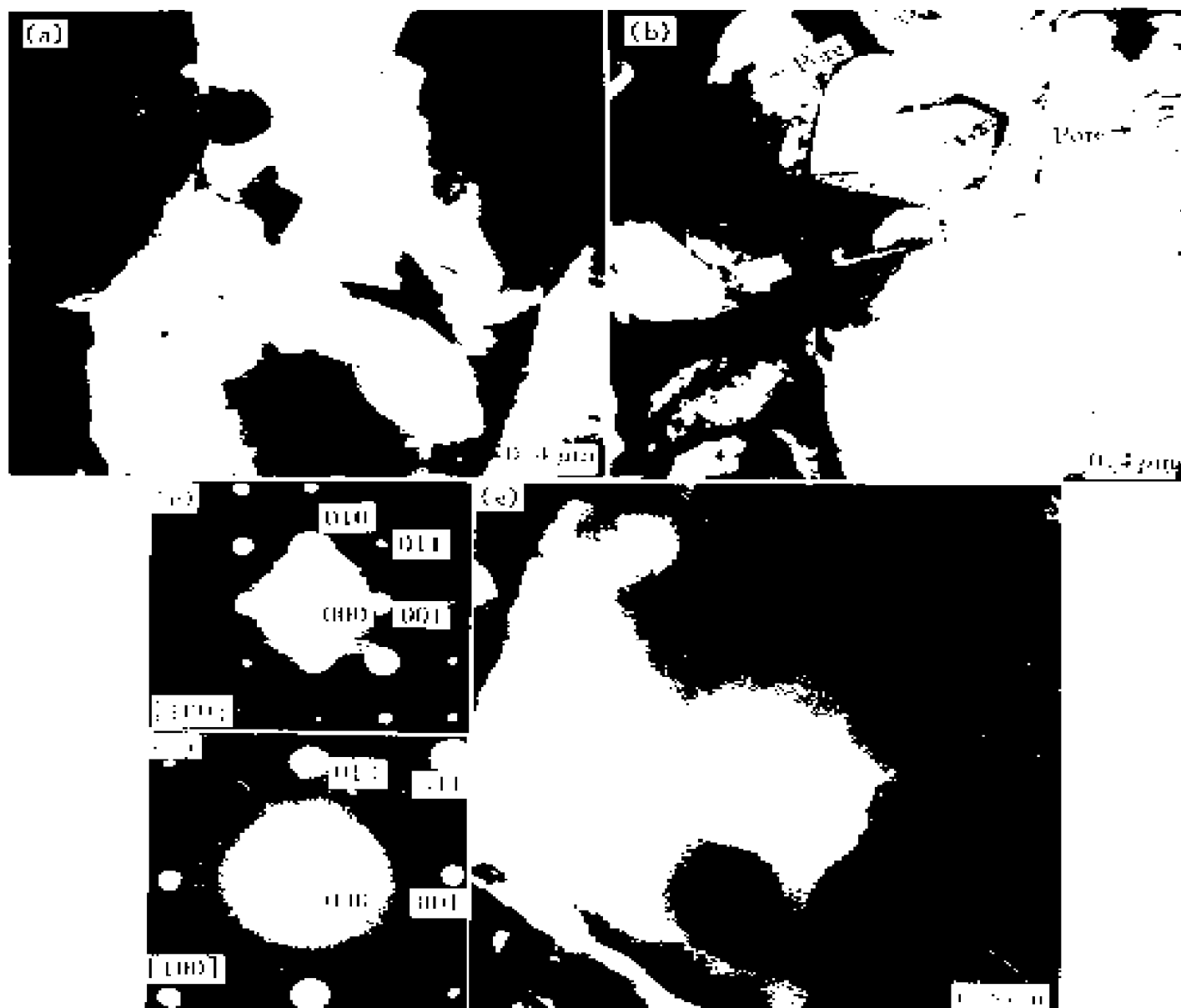
Mechanical properties of the composite, TNS, are shown in Table 2. Mechanical properties of TN1 and TN2 composites related to TNS are also listed for comparison. Compared with Sample TN1, Sample TNS has lower flexural strength at room temperature ( $\sigma_{RT}$ ) but much higher flexural strength at 1000 °C ( $\sigma_{1000\text{ }^{\circ}\text{C}}$ ); while compared with Sample TN2, Sample TNS has higher mechanical properties. It can be known from Table 2 that using nickel base superalloy instead of nickel has a significant effect on improving the mechanical properties of (Nb, Ti) C-Ni composite, especially the flexural strength at elevated temperature.

**Table 2 Mechanical properties of (Nb, Ti) C-nickel base superalloy composite**

Sample	$\sigma_{RT}/$ MPa	$\sigma_{1000\text{ }^{\circ}\text{C}}/$ MPa	$K_{IC}/$ (MPa $\cdot$ m <sup>1/2</sup> )	HRA
TN1	1630 $\pm$ 190	320 $\pm$ 45	18.8	85.5
TN2	1250 $\pm$ 120	210 $\pm$ 55	12.5	82.0
TNS	1380 $\pm$ 158	508 $\pm$ 50	12.8	84.0

### 3.2 Microstructure of composite

In order to explain the reasons for the reinforcement of using nickel base superalloy instead of nickel, Sample TNS was observed with TEM. It was found that the microstructure of the sample was very complicate and the size of the (Nb, Ti) C grain in the sample was small (about 2  $\mu$ m). Fig. 1(a) is a TEM micrograph of (Nb, Ti) C grains containing dislocations. Fig. 1(b) shows a few (Nb, Ti) C grains containing micropores. Occasionally, (Nb, Ti) C grain consisting of surrounding structure was observed (indicated as an arrow in Fig. 1(b)). The surrounding structure was analysed with selected



**Fig. 1 TEM micrographs of (Nb, Ti) C grains**

- (a) —Containing dislocations; (b) —Containing pore and surrounding structure;  
 (c) —SAED pattern of center of surrounding structure;  
 (d) —SAED pattern of surrounding layer; (e) —A large (Nb, Ti) C grain containing many subgrains

area electron diffraction. The analysis results shown in Fig. 1(c) and (d) indicated that the crystal structure of the center and the surrounding layer of the surrounding structure were both similar to TiC structure. Similar surrounding structure, as mentioned in part one, was not found in TN2 and TNY composites but in TN1 composite. Therefore, the formation of the surrounding structure is independent of the preparation method of (Nb, Ti) C powder. In the TEM observation, it was also found that some large (Nb, Ti) C grains containing many subgrains that

were not found in other composites (Fig. 1(e)). The formation mechanism of the subgrains was not clear.

#### 4 DISCUSSION

Regardless of the influence of the microstructure (size of grain, pore), generally, the flexural strength of the ceramic-metal composite depends on three elements, namely, the strength of metal phase, the strength of ceramic grain and binding force between metal phase and

ceramic grains.

At room temperature, the flexural strength of (Nb,Ti)C-Ni composite system depends mainly on binding force between metal phase and ceramic grains<sup>[12]</sup>. If there are pores in the system, the pores will lead to stress concentration, produce microcracks and cause fracture finally under applied stress. Therefore, the fewer pores in the system, the better flexural strength of the system. For the two (Nb,Ti)C powder preparation methods, i. e., arc melting and thermal explosion, because of larger temperature gradients and higher heating and cooling rates experience during thermal explosion, higher defect (pore) concentrations are likely to be present to thermal explosion product. Although most of the pores are illuminated in the HIP procedure, but a little part of the pores, especially the closed pores are difficult to illuminate. On the other hand, compared with Sample TN1, Sample TNS has fewer (Nb,Ti)C grains containing surrounding structure which can improve the binding between ceramic phase and metal phase. Therefore, Sample TNS has lower flexural strength at room temperature than that of TN1. While compared with Sample TN2, firstly, Sample TNS has a few (Nb,Ti)C grains containing surrounding structure which can increase the binding force between ceramic phase and metal phase; secondly, some (Nb,Ti)C grains in it contains subgrains (Fig. 1(e)), the surface energy of grain boundary increases with the formation of subgrain and it becomes more difficult to make split between ceramic grain and metal binder phase, and finally, it can be known from Fig. 1(a) that dislocations are difficult to run through the grain boundary because of grain boundary and different crystal orientation of grains between the grain boundary, namely, the grain boundary may prevent the movement of dislocation and has reinforcement effect. Therefore, Sample TNS has higher flexural strength than that of Sample TN2.

At elevated temperature (~ 1 000 °C), the metal phase is in viscous state and its flexural strength decreases rapidly, therefore, under this circumstance the flexural strength of the system

depends mainly on metal phase. On the other hand, the dependence of the rate of creep deformation of a material on porosity follows the relation  $\dot{\epsilon} \sim (1 - P^{2/3})^{-1}$ , where  $\dot{\epsilon}$  and  $P$  are the rate of creep deformation and porosity respectively, that is to say, the pores will accelerate the rate of creep deformation of the composite. Therefore, Sample TN2 has the lowest flexural strength at 1 000 °C. Although Sample TN1 has few pores, it still has lower flexural strength because of poor strength of metal phase at 1 000 °C. While in Sample TNS, as we know, because of the reinforcement effect of other elements on Ni, such as solution strengthening of Cr, W and Mo, precipitation strengthening of Ta, Al and Ti and so on, the strength of nickel base superalloy is much higher than that of Ni. The flexural strength of Sample TNS at 1 000 °C is much high even there are a few pores in it.

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