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Fracture behavior and microstructure of as-cast NiTi shape memory alloy

JIANG Shu-yong[1], ZHANG Yan-qiu[1], FAN Hong-tao[2]

1. Industrial Training Centre, Harbin Engineering University, Harbin 150001, China;
2. College of Materials Science and Chemical Engineering, Harbin Engineering University, Harbin 150001, China

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Abstract: The as-cast ingot of equiatomic nickel−titanium shape memory alloy (NiTi SMA) was prepared by vacuum consumable arc melting. The tensile tests and the compressive tests with respect to as-cast NiTi SMA were performed to study its mechanical properties of fracture. The microanalysis of as-cast NiTi SMA as well as its fractured samples was performed so as to better understand microstructure evolution and fracture behavior of NiTi SMA. Under tensile loading, the as-cast NiTi SMA shows higher plasticity and is characterized by ductile fracture at 750 °C, but it demonstrates poorer plasticity and is characterized by cleavage fracture as well as transcrystalline fracture at room temperature and −100 °C. Under compressive loading at −100 °C, the as-cast NiTi SMA is characterized by shear fracture where the normal to the shearing fracture surface inclines about 45° to the compressive axis, and belongs to cleavage fracture where the cracks expand via transcrystalline fracture.

Key words: NiTi alloy; shape memory alloy; as-cast NiTi; microstructural evolution; fracture behavior

1 Introduction

Nickel−titanium shape memory alloy (NiTi SMA) is widely used in engineering fields because it possesses shape memory effect and superelasticity, which are attributed to thermo-elastic martensite phase transformation of NiTi SMA [1]. The application of shape memory effect and superelasticity of NiTi SMA to engineering fields is considerably dependent on the phase transformation temperature. It is well known that NiTi SMA shows excellent superelasticity only above the austenite transformation finish temperature A_f, and exhibits full shape recovery when heated to the temperature above A_f. The transformation temperatures of NiTi SMA are extremely sensitive to a small variation in the Ni or Ti content. As for NiTi SMA having greater than 55.0% Ni (mass fraction), a 1% deviation in Ni (or Ti) content will result in approximately a shift of 100 °C in transformation temperatures [2]. FRENZEL et al [3] demonstrated that the increase of Ni content in NiTi SMA decreases the phase transformation temperatures as well as the width of thermal hysteresis and the heat of transformation. Therefore, the strict requirement on any melting by tightly controlling the ratio of Ni to Ti so as to meet the required tolerance in the transformation temperatures plays a significant role in successful application of NiTi SMA to engineering fields [4].

The processes to melt NiTi SMA are commonly made up of vacuum induction melting (VIM) and vacuum arc melting (VAM) which includes consumable arc melting and non-consumable arc melting [5,6]. VIM contributes to homogeneity of chemical composition of NiTi SMA, but it needs using graphite crucible which leads to the reaction between Ti and C. As a result, titanium carbide (TiC) is formed in NiTi SMA during melting and solidification, which results in the increase of Ni content of the matrix of the alloy and thus decreases the martensite transformation temperature [7,8]. VAM needs no graphite crucible and thus causes little contamination of NiTi SMA from the crucible, but it usually leads to less homogeneous chemical composition of NiTi SMA ingot and requires multiple re-melting [9].

The inhomogeneous chemical composition of as-cast NiTi SMA ingot inevitably leads to uneven microstructure and thus has an influence on the mechanical properties of NiTi SMA. Cold working and heat treatment play important roles in guaranteeing the final mechanical properties of NiTi SMA [10,11].

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Corresponding author: JIANG Shu-yong; Tel: +86-451-82519706; E-mail: jiangshy@sina.com
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Some researchers investigated the microstructures and the mechanical properties of as-cast NiTi SMA, which lays the foundations for the subsequent thermal processing of as-cast NiTi SMA. KHAMEI and DEHGHANI [12] established the constitutive equation of as-cast NiTi SMA with homogenized annealing at a high temperature which complies with the Zener-Hollomon parameter and the fact is obtained that NiTi SMA exhibits obviously dynamic recrystallization under quasi-static compression deformation at the temperatures ranging from 950 to 1050 °C [13]. MORAKABATI et al [14] studied the hot tensile properties of as-cast NiTi SMA at a strain rate of 0.1 s^{-1} at the elevated temperatures and found that as-cast NiTi SMA shows a maximum hot ductility at the temperatures ranging from 750 to 1000 °C and dynamic recrystallization at the temperatures ranging from 800 to 1000 °C, where the tensile fractograph of NiTi SMA belongs to a typical ductile rupture. RAGHAVENDRA et al [15] investigated the tensile fracture of the annealed NiTi SMA under quasi-static and dynamic loading at various temperatures and discovered that NiTi SMA shows quasi-cleavage fracture at a low temperature of −196 °C as well as the typical ductile fracture at high temperatures (200−400 °C), and the mixture of the two at room temperature [15].

This study is aimed at understanding fracture and microstructure of as-cast NiTi SMA which experiences no heat treatment or plastic working history. So far, fracture behaviour and microstructure of as-cast NiTi SMA have been reported under tensile and compressive loading in the few literatures.

2 Experimental

The ingot of NiTi SMA with nominal compositions of 50.0%Ni−50.0%Ti (mole fraction) was prepared in a water-cooled copper crucible by means of vacuum consumable arc melting, where the raw materials used were the Ni powder with the purity of 99.97% and the sponge Ti with the purity of 99.95%. The Ni powder and sponge Ti were mixed according to the equiatomic proportion, and then were made into the electrode bulk by means of sintering at high temperature and high pressure in the vacuum atmosphere. The electrode bulk was melted by means of vacuum consumable arc melting and then was cast into the ingot of NiTi SMA with the diameter of 80 mm and the height of 80 mm, as shown in Fig. 1.

The tensile samples and the compressive samples were prepared from as-cast ingot of NiTi SMA by means of machining. The dimensions of the tensile samples are shown in Fig. 2. The compressive samples possess the diameter of 6 mm and the height of 9 mm. The tensile tests and compressive tests with respect to the samples were carried out on INSTRON−5500R at the strain rate of 0.001 s in order to obtain the mechanical properties of as-cast NiTi SMA. Metallographic investigation of as-cast NiTi SMA as well as the NiTi SMA samples after the tensile tests was performed. Specimens for microstructure observation were cut from as-cast NiTi SMA and the NiTi SMA samples after the tensile and compressive tests. All specimens were etched in a solution containing HF, HNO_3 and H_2O whose volume ratio was 1:4:5 and then studied by means of OLYMPUS metallographic microscope. The fracture surface of the tensile samples was observed by QUANTA200 scanning electron microscope (SEM).

Fig. 1 Photo of as-cast ingot of NiTi SMA

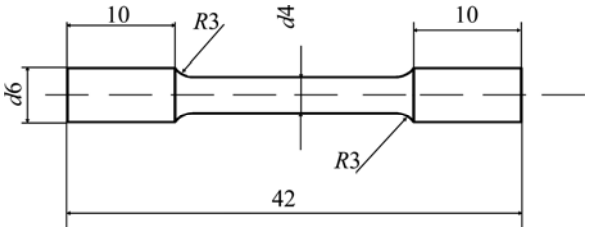

Fig. 2 Schematic diagram of dimensions of NiTi SMA tensile sample (unit: mm)

3 Results and discussion

3.1 Microstructure of as-cast NiTi SMA

Figure 3 shows the microstructures of as-cast NiTi SMA, from which it can be seen that the microstructure of the as-cast NiTi SMA which consists of dendritic grains and equiaxed grains is very inhomogeneous. Figure 4 shows the binary phase diagram of the NiTi alloy [1]. NiTi SMA with equiatomic ratio belongs to β parent phase at the high temperature and possesses BCC structure. When cooling to 1090 °C, the structure of the parent phase of TiNi alloy is transited to austenite with $B2$ type ordered structure. With the continuous decrease of temperature, the $B2$ austenite is transformed into the

$B19'$ martensite. The space group of the $B19'$ martensite is $P2_1/m$ with a monoclinic unit cell. Therefore, theoretically, the microstructure of as-cast NiTi SMA at room temperature should be made up of $B2$ austenite or $B19'$ martensite, or a mixture of the two ones. However, in fact, the as-cast NiTi SMA consists of $B19'$ martensite, $B2$ austenite and Ti_2Ni phase simultaneously at room temperature, which will be reported in another literature. The phenomenon is attributed to the segregation of as-cast NiTi SMA during crystallization.

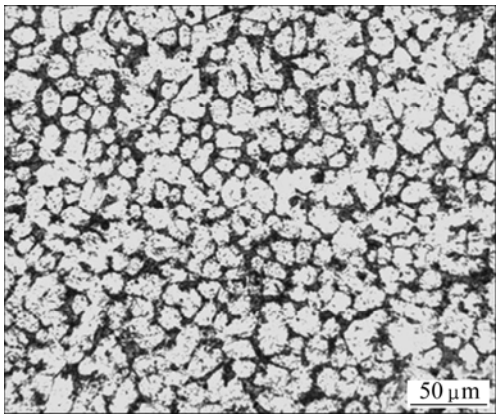

Fig. 3 Optical microstructure of as-cast NiTi SMA ingot

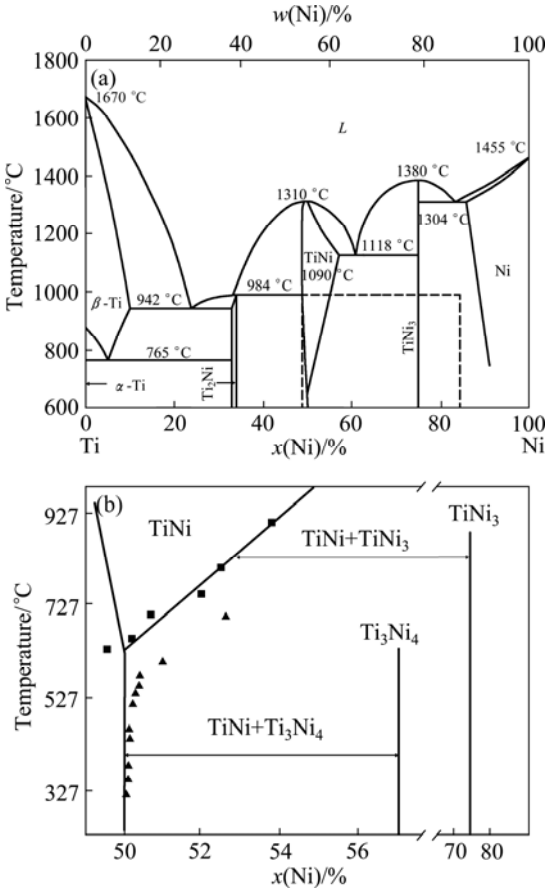

Fig. 4 Phase diagram of NiTi alloy: (a) Metastable Ti_3Ni_4 phase; (b) Phase equilibrium between TiNi and Ti_3Ni_4 phases added in dashed zone of (a)

3.2 Tensile fracture of as-cast NiTi SMA

3.2.1 Tensile fracture at 750 °C

Figure 5 indicates the fractograph of as-cast NiTi SMA after tensile fracture at 750 °C. It can be obviously seen from Fig. 5 that NiTi SMA shows a great macroscopic plastic deformation before tensile fracture. The fractograph looks like blade and is characterized by orientation, which belongs to the typical ductile rupture. In order to better understand the deformation law of NiTi SMA under tensile loading at high temperature, the corresponding tensile fractured sample in Fig. 5 is divided into three zones including zone A, zone B and zone C against the direction perpendicular to the blade along the longitudinal cross-section, which is shown in Fig. 6. The corresponding microstructures of NiTi SMA in the three zones are illustrated in Fig. 7. Zone A belongs to the severely plastic deformed one in which the grains of NiTi SMA are considerably elongated along the tensile direction, and thus shows the obvious feature of fibrous tissue. Zone C belongs to the slightly plastic deformed one in which no obvious plastic deformations takes place and the microstructure shows a characteristic of the equiaxed grains. Zone B belongs to the transient one between zone A and zone B, where a certain plastic deformation occurs.

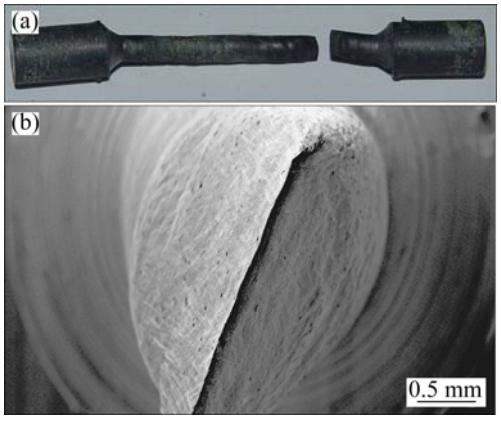

Fig. 5 Fractograph of NiTi SMA after tensile fracture at 750 °C: (a) Fractured sample; (b) SEM image

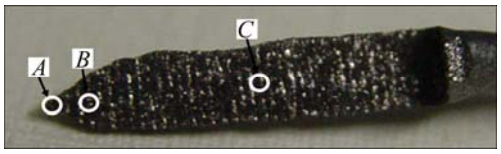

Fig. 6 Schematic diagram of different zones at tensile fractured sample of NiTi SMA at 750 °C

3.2.2 Tensile fracture at room temperature

Figure 8 demonstrates the fractograph of NiTi SMA after tensile fracture at room temperature. The slight plastic deformation of NiTi SMA occurs before tensile fracture and the fractograph of NiTi SMA shows the

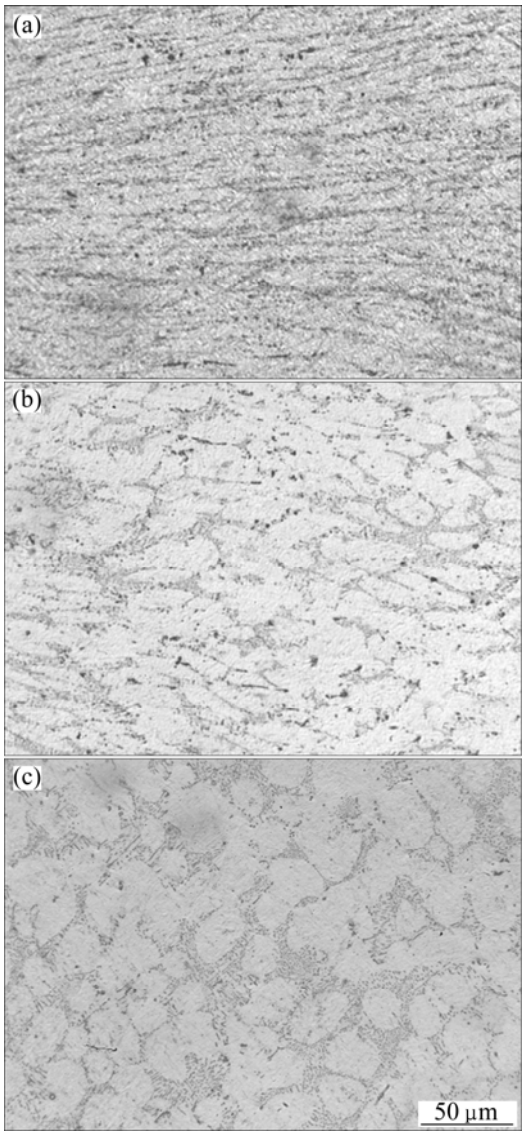

Fig. 7 Microstructures of different zones in tensile fractured sample of NiTi SMA at 750 °C: (a) Zone *A* in Fig. 6; (b) Zone *B* in Fig. 6; (c) Zone *C* in Fig. 6

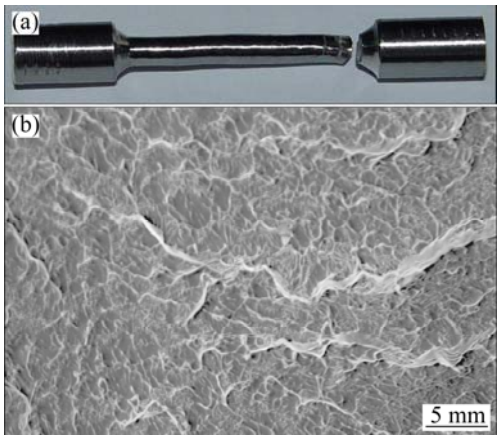

Fig. 8 Fractograph of NiTi SMA after tensile fracture at room temperature: (a) Fractured sample; (b) SEM image

brittle fracture feature. Furthermore, the fractograph consists of cleavage plane and tear ridge around the cleavage plane. Therefore, the fractograph of NiTi SMA is typical of cleavage fracture. It can be obviously seen from the microstructure of NiTi SMA sample after tensile fracture in Fig. 9 that the crack of NiTi SMA expands by means of transcrystalline fracture.

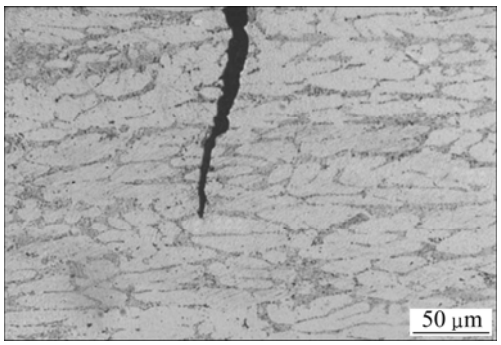

Fig. 9 Microstructure of tensile fractured sample of NiTi SMA at room temperature

3.2.3 Tensile fracture at −100 °C

Figure 10 demonstrates the fractograph of NiTi SMA sample after tensile fracture at −100 °C. The fractograph of NiTi SMA is also characterized by the typical cleavage fracture, but is very different from that at room temperature. NiTi SMA belongs to the full martensite structure at −100 °C which is far less than martensite transformation finish temperature (M_f), so the fractograph of NiTi SMA at −100 °C possesses a characteristic of fracture surface of martensite. However, the microstructure of NiTi SMA is a mixture of austenite and martensite at room temperature, so the fractograph is a product of fracture surface of austenite and martensite. Furthermore, the fractograph of NiTi SMA in Fig. 10 may be attributed to the fracture of microstructure of dendritic grains. Figure 11 shows the microstructure of NiTi SMA sample after tensile fracture at −100 °C,

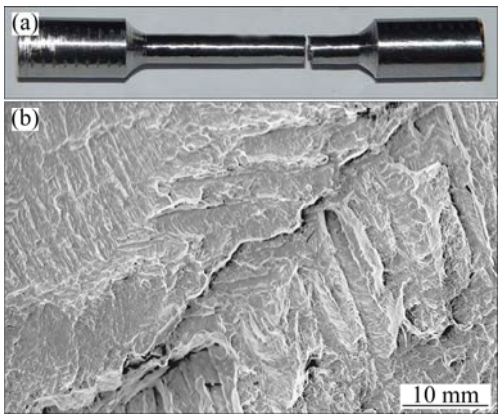

Fig. 10 Fractograph of NiTi SMA after tensile fracture at −100 °C: (a) Fractured sample; (b) SEM image

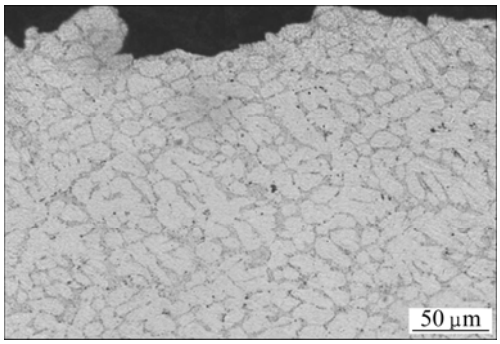

Fig. 11 Microstructure of tensile fractured sample of NiTi SMA at −100 °C

where the crack still expands by means of transcrystalline fracture.

3.3 Compressive fracture of as-cast NiTi SMA

The compressive deformation of as-cast NiTi SMA samples at −100 °C leads to fracture of the compressed sample at the strain rate of 0.001 s^{-1}, as shown in Fig. 12. It can be seen from Fig. 12 that the fractured sample is characterized by shear fracture and the angle between the normal to the shearing fracture surface and the compressive axis is about 45°. The fractograph of the corresponding fractured sample shown in Fig. 12 has a characteristic of river pattern containing cleavage plane and tear ridge and thus belongs to cleavage fracture. Furthermore, the microstructure of the compressive fractured sample of as-cast NiTi SMA at −100 °C in Fig. 13 indicates that the cracks expand by means of transcrystalline fracture.

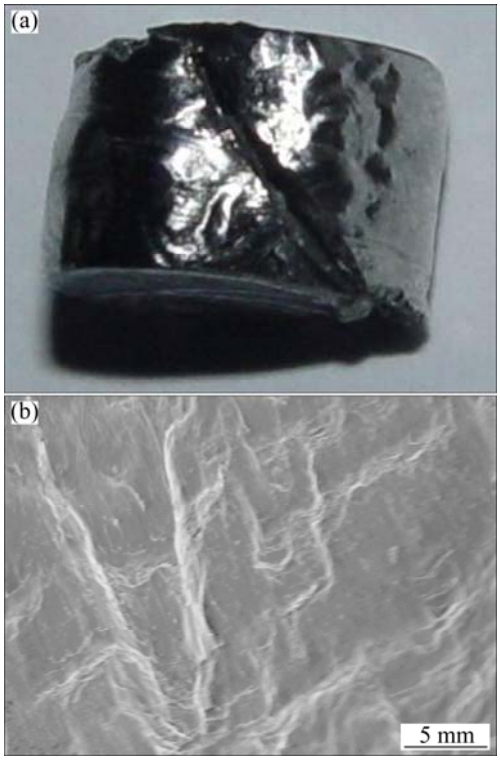

Fig. 12 Fractographs of fractured samples of as-cast NiTi SMA under compression at −100 °C: (a) Fractured sample; (b) SEM image

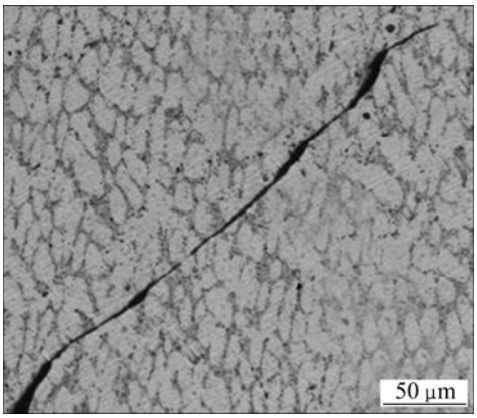

Fig. 13 Microstructure of compressed samples of as-cast NiTi SMA at −100 °C

4 Conclusions

1) The as-cast NiTi SMA prepared by vacuum consumable arc melting shows inhomogeneous microstructure, which consists of dendritic grains and equiaxed grains, and is a mixture of *B*19′ martensite, *B*2 austenite and Ti_2Ni phase at room temperature. Therefore, multiple remelting is an approach to obtain NiTi SMA with uniform microstructure.

2) Under tensile loading, the as-cast NiTi SMA is characterized by ductile fracture at 750 °C, but cleavage fracture at room temperature as well as −100 °C. Furthermore, the crack expands by means of transcrytalline fracture during fracture of as-cast NiTi SMA at room temperature as well as −100 °C.

3) Under compressive loading at −100 °C, the fractured sample of the as-cast NiTi SMA is characterized by shear fracture and the angle between the normal to the shearing fracture surface and the compressive axis is about 45°. The fractograph of the as-cast NiTi SMA has a characteristic of river pattern containing cleavage plane and tear ridge, and thus belongs to cleavage fracture. The cracks expand by means of transcrystalline fracture.

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铸态镍钛形状记忆合金的断裂行为及显微组织

江树勇[1], 张艳秋[1], 范红涛[2]

1. 哈尔滨工程大学 工程训练中心，哈尔滨 150001；
2. 哈尔滨工程大学 材料科学与化学工程学院，哈尔滨 150001

摘　要：通过真空自耗电极熔炼法制备等原子比镍钛形状记忆合金。为了研究其断裂力学性能, 进行铸态镍钛形状记忆合金的拉伸和压缩实验。为了更好地理解镍钛形状记忆合金的组织演变及断裂行为，分析铸态镍钛形状记忆合金及其断裂样品的显微组织。在拉伸加载下，镍钛形状记忆合金在 750 °C 时具有较高的塑性，表现为韧性断裂，但在室温和−100 °C 时表现出较差的塑性，具有解理断裂和穿晶断裂的特征。在−100 °C 的压缩加载下，铸态镍钛形状记忆合金发生剪切断裂，剪切断裂面法线与压缩轴呈 45°，具有解理断裂的特征，裂纹经由穿晶断裂而扩展。

关键词：镍钛合金；形状记忆合金；铸态镍钛；组织演变；断裂行为

(Edited by LI Xiang-qun)