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Trans. Nonferrous Met. Soc. China 22(2012) 90−96
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Microstructure evolution and deformation behavior of as-cast NiTi shape memory alloy under compression

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Received 25 February 2011; accepted 7 September 2011

Abstract: The as-cast ingot of equiatomic nickel-titanium shape memory alloy (SMA) was prepared via vacuum consumable arc melting. Differential scanning calorimetry (DSC), X-ray diffraction (XRD) analysis, energy dispersive X-ray (EDX) measurement and microanalysis were adopted in order to understand the microstructure evolution and deformation behavior of the as-cast NiTi SMA under compression at various strain rates and temperatures. The microstructures of as-cast NiTi SMA composed of dendritic grains and equiaxed grains are inhomogeneous and show segregation. The as-cast NiTi SMA consists of B19′ martensite, B2 austenite and Ti_2Ni phase simultaneously at room temperature. The as-cast NiTi SMA is sensitive to strain rate under compression at high temperature, at which NiTi SMA is characterized by dynamic recrystallization at strain rates of 0.1 and 0.01 s^{-1}, but by dynamic recovery at strain rate of 0.001s^{-1}. The strain rates have little influence on the true stress—strain curves as well as microstructure of NiTi SMA at room temperature and −100 °C.

Key words: as-cast NiTi; microstructure evolution; deformation behavior; dynamic recrystallization; shape memory alloy

1 Introduction

Nickel-titanium shape memory alloy (NiTi SMA) is widely used in engineering fields because of its shape memory effect as well as superelasticity. Shape memory effect of NiTi SMA refers to its ability to remember the shape at a certain state. Specifically, NiTi SMA is imparted to a certain shape at the austenite phase and then is transformed into martensite phase, where NiTi SMA undergoes a certain plastic deformation at martensite phase and then is able to go back to the previous shape at the austenite phase on heating. Shape memory effect of NiTi SMA is attributed to reversible thermoelastic martensite transformation. Reversibility of martensite transformation refers to austenite being able to transform into martensite on cooling as well as martensite being able to transform into austenite on heating. Superelasticity of NiTi shape memory alloy refers to its ability to recover its original shape along with the release of stress after NiTi SMA has been deformed beyond its elastic limit by external mechanical stress. The mechanism of superelasticity of NiTi SMA results from stress-induced martensite transformation as well as reversible transformation of martensite.

Stress and temperature are the two external factors that have an important influence on the shape memory effect as well as superelasticity of NiTi SMA. Furthermore, the loading modes and strain rates have an important influence on the mechanical behavior of NiTi SMA. Over the last decades, many scholars studied the mechanical behavior of NiTi SMA under various loading modes and strain rates. Under uniaxial tensile loading, NiTi SMA usually exhibits the localized Lüders-like deformation behavior characterized by the occurrence of a stress plateau in the stress—strain curve, which is conventionally attributed to the stress-induced martensite transformation of NiTi SMA in the austenite state [1, 2]. SHAW and KYRIAKIDES [3] demonstrated that NiTi SMA strip in an austenitic phase shows a martensitic band nucleation at a critical stress level under uniaxial tension by means of morphology observation experiments, where the martensitic band inclines at 55° to the axis of loading. LI and SUN [4] observed the nucleation, growth and coalescence of the spiral martensite band in a NiTi SMA tube of the austenite phase under uniaxial tension via optical microscopy, where the spiral martensite band inclines at 57° to the

Foundation item: Project (51071056) supported by the National Natural Science Foundation of China; Project (HEUCFR1132) supported by the Fundamental Research Funds for the Central Universities of China
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DOI: 10.1016/S1003-6326(11)61145-X

axis of loading. PENG et al [5] and MAO et al [6] also obtained the spiral martensite band in a NiTi SMA tube of austenite phase under uniaxial tension via optical microscopy, but the angles of the spiral martensite band to the axis of loading are slightly different, the former is 60° and the latter is 58°. However, NG and SUN [7] discovered the Lüders-like deformation in a NiTi SMA tube of R-phase under tensile loading, which is attributed to the transformation from R-phase to martensite based on stress inducing. NiTi SMA also demonstrates the Lüders-like deformation behaviour due to the martensite reorientation in the martensite state under uniaxial tensile loading [8]. So far, the Lüders-like deformation behaviour has never been observed in the other loading modes such as compression and shear [9−11]. In particular, the stress—strain curve of NiTi SMA under compression obviously shows an asymmetry characteristic with respect to that under tension [12−14]. GALL et al [12] studied the deformation behavior of single crystal and polycrystalline NiTi SMA with the same chemical composition in an austenite state under tension and compression. SEHITOGLU et al [13] investigated the compressive response of single crystal NiTi SMA in an austenite state where the deformation behavior of the single crystal NiTi SMA is dependent on the crystallographic orientation under compression. LIU et al [14] studied the asymmetry of deformation behavior of polycrystalline NiTi shape memory alloy in a martensite state under tension and compression, and thought that the martensite deformation under tension may be mainly associated with the interfacial migration between two adjacent martensite plates, but it was mainly related to the generation and movement of dislocations under compression. Many researchers have investigated the influence of the strain rates on the deformation behavior of NiTi SMA under compressive loading [15−17]. The stress—strain curves of martensitic NiTi SMA are insensitive to the strain rates under quasi-static compression [15]. However, the deformation behavior of NiTi SMA at high strain rates is different from that at low strain rates. CHEN et al [16] found that the stress—strain curves of austenitic NiTi SMA exhibit closed stress hysteresis loops under quasi-static compression, but show open stress hysteresis loops under dynamic compression. NEMAT-NASSER et al [17] discovered that the austenitic NiTi SMA exhibits direct plastic deformation for dislocation slip rather than stress-induced martensitic transformation at very high strain rate, e.g., 17000 s^{-1}.

In this study, the emphasis is laid on the influence of segregation on the microstructure evolution and deformation behavior of as-cast NiTi SMA under compression at various temperatures and strain rates. The as-cast NiTi SMA undergoes no heat treatment or plastic working history. So far, few literatures have reported the deformation behavior of the as-cast NiTi SMA under compression.

2 Experimental

The as-cast ingot of NiTi SMA with nominal composition of 50.0% Ni−50.0% Ti was prepared in a water-cooled copper crucible by means of vacuum consumable arc melting. Differential scanning calorimetry (DSC) test was performed at heating and cooling rates of 20 °C/min in order to obtain the transformation temperatures of as-cast NiTi SMA. X-ray diffraction (XRD) analysis was used to acquire the phase structure of as-cast NiTi SMA. The samples of as-cast NiTi SMA with size of *d*6 mm×9 mm were fabricated by means of electro-discharge machining (EDM) and then were used for the compression tests at various strain rates as well as temperatures. The compressed samples were cut along the axial direction so as to observe the microstructures. The cutting and microstructure observation directions of the samples are shown in Fig.1. The microstructures of the as-cast NiTi SMA and the compressed samples were observed by means of optical microscopy and scanning electron microscopy (SEM). All the specimens were etched in a solution of 10% HF+40% HNO_3+50% H_2O. The SEM is equipped with an energy dispersive X-ray (EDX) analysis unit for chemical microanalysis. EDX measurements were used for determining the compositions of the matrix and the secondary phases in as-cast NiTi SMA.

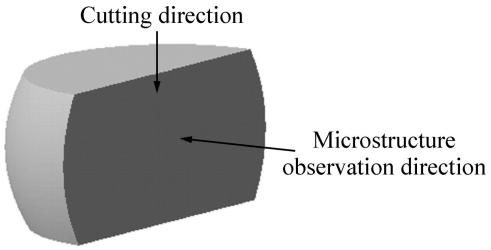

Fig. 1 Schematic diagram of cutting and microstructure observation directions of samples

3 Results and discussion

3.1 Microstructure of as-cast NiTi SMA

Figure 2 shows the microstructure of the as-cast NiTi SMA by means of SEM. It is evident from Fig. 2 that the microstructures of the as-cast NiTi SMA which consists of dendritic grains and equiaxed grains are very inhomogeneous and even show severe segregation. Figure 3 demonstrates the EDX spectra of the as-cast NiTi SMA, which reveals that the matrix phase of the as-cast NiTi SMA is NiTi and the Ti_2Ni phase distributes

among the NiTi matrix phases. The occurrence of the Ti_2Ni phase causes the NiTi matrix to be rich in Ni.

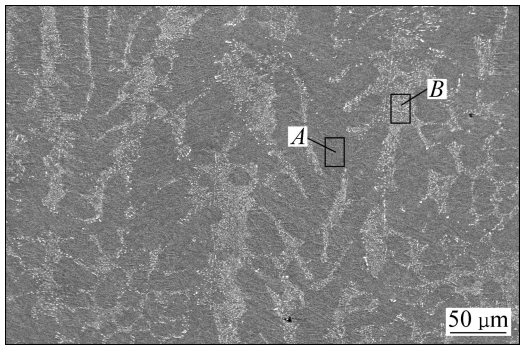

Fig. 2 SEM micrograph of as-cast NiTi SMA

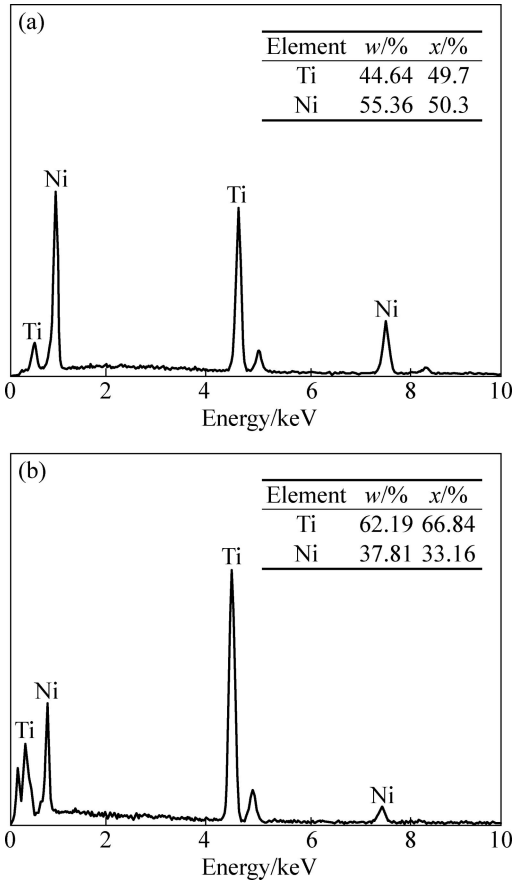

Fig. 3 EDX spectra of as-cast NiTi SMA: (a) Area *A* in Fig. 2; (b) Area *B* in Fig. 2

3.2 DSC analysis of as-cast NiTi SMA

Figure 4 shows the DSC curve of the as-cast NiTi SMA. It can be seen from the DSC curve that the transformation temperatures of the as-cast NiTi SMA are M_s=34.2 °C, M_f=19.2 °C, A_s=45.8 °C, A_f=65.7 °C and which are obviously lower than the results in Ref. [18]. The reason is that the Ti_2Ni phase is formed in NiTi SMA during melting and solidification, which leads to the increase of Ni concentration of the matrix of the alloy and thus decreases the phase transformation temperatures.

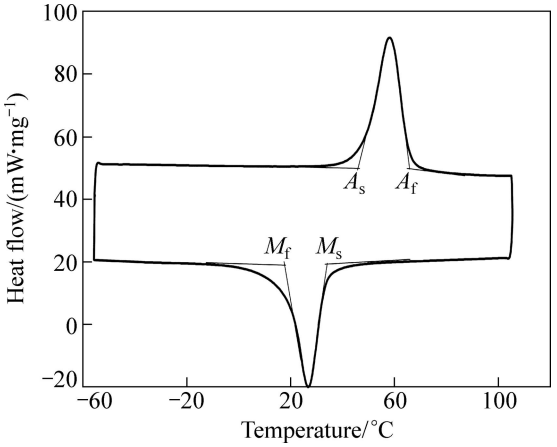

Fig. 4 DSC curve of as-cast NiTi SMA

3.3 XRD analysis of as-cast NiTi SMA

The XRD diagram of the as-cast NiTi SMA is illustrated in Fig. 5. It can be obviously seen that the as-cast NiTi SMA consists of B19′ martensite phase, B2 austenite phase and Ti_2Ni phase simultaneously at room temperature, which is in accordance with the results from the DSC test. Martensite and austenite coexist because the martensite finish temperature (M_f) of the as-cast NiTi SMA is lower than the room temperature, which leads to incomplete transformation of as-cast NiTi SMA from austenite to martensite at room temperature.

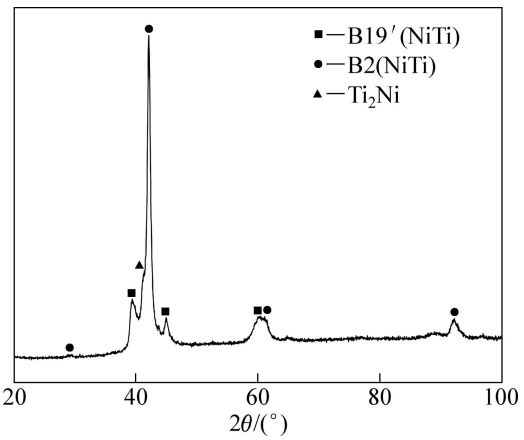

Fig. 5 XRD pattern of as-cast NiTi SMA

3.4 Deformation behavior of NiTi SMA under compression

3.4.1 Deformation behavior of NiTi SMA at high temperature

Figure 6 shows the true stress—strain curves of NiTi SMA samples under compression at various strain rates at 750 °C. Figure 7 shows the microstructures of the involved compressed samples. It can be obviously seen from Fig. 5 that NiTi SMA is sensitive to strain

rates at high temperature. It is evident from the true stress—strain curve at strain rate of 0.1 s^{-1} that NiTi SMA is obviously characterized by dynamic recrystallization. Before the critical deformation degree, working hardening of NiTi SMA leads to the rapid increase of stress, but after the critical deformation degree, dynamic recrystallization of NiTi SMA leads to the advantage of softening over hardening, so the stress of NiTi SMA rapidly goes down. However, the microstructure of the corresponding compressed sample consists of plenty of equiaxed grains and shows hardly obvious dynamic recrystallization. The above phenomenon indicates that the deformation velocity is so rapid that the crystal nucleus formed during dynamic recrystallization are unable to grow in time, so dynamic recrystallization can be hardly observed via metallographic microscope under low magnification, which should be further validated by means of the involved experiments. As for the true stress—strain curve at strain rate of 0.01 s^{-1}, NiTi SMA also demonstrates the dynamic recrystallization features. The microstructure of the corresponding compressed sample shows obviously dynamic recrystallization characteristics because the grains are more considerably refined than the as-cast NiTi SMA. Under compressive deformation at strain rate of 0.001 s^{-1}, NiTi SMA shows dynamic recovery rather than dynamic recrystallization. NiTi SMA has a significant characteristic of grain elongation during dynamic recovery.

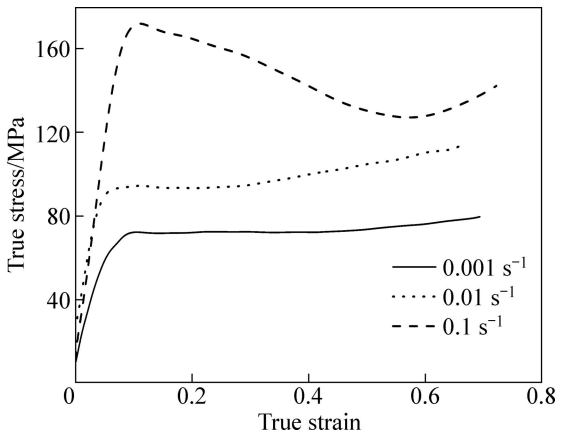

Fig. 6 True stress—strain curves of as-cast NiTi SMA at 750 °C

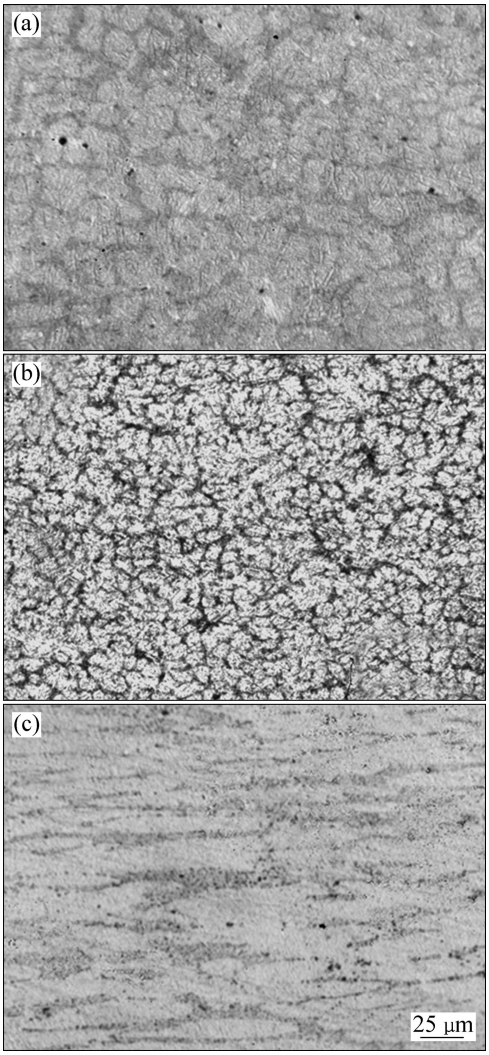

Fig. 7 OM images of compressed samples of as-cast NiTi SMA at 750 °C at different strain rates: (a) 0.1 s^{-1}; (b) 0.01 s^{-1}; (c) 0.001 s^{-1}

3.4.2 Deformation behavior of NiTi SMA at room temperature

Figure 8 indicates the true stress—strain curves of NiTi SMA samples under compression at various strain rates at room temperature. Figure 9 shows the microstructures of the corresponding compressed samples. The true stress—strain curves of the as-cast NiTi SMA samples under compression at various strain rates at room temperature show that the strain rates have little influence on the true stress—strain curves of as-cast NiTi SMA. Furthermore, compared with the original as-cast microstructure, the microstructures of NiTi SMA after compress deformation at room temperature still

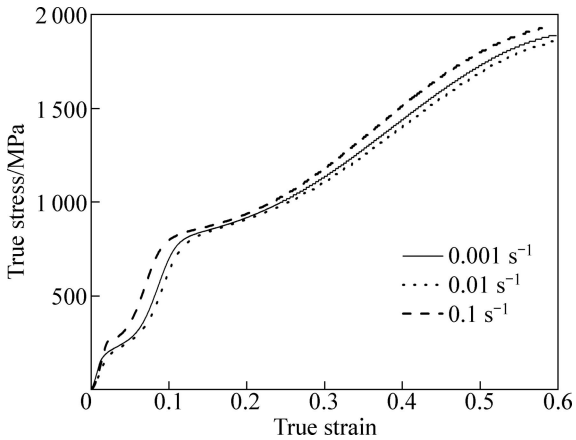

Fig. 8 True stress—strain curves of as-cast NiTi SMA at room temperature

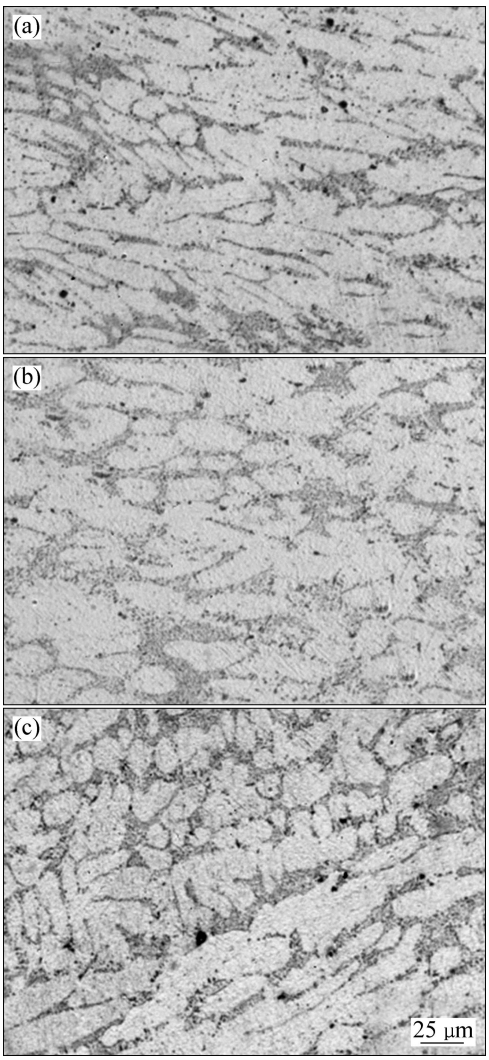

Fig. 9 OM images of compressed samples of as-cast NiTi SMA at room temperature at different strain rates: (a) 0.1 s^{-1}; (b) 0.01 s^{-1}; (c) 0.001 s^{-1}

possess a lot of dendritic grains. Because the as-cast NiTi SMA contains B2 austenite phase and B19′ martensite phase, the deformation behavior of NiTi SMA under compression is more complex. NiTi SMA generally experiences four stages, namely elastic deformation of austenite and martensite, stress-induced martensitic transformation along with martensite variant reorientation and detwinning, elastic deformation of stress-induced martensite and reoriented martensite, and plastic deformation of stress-induced martensite and reoriented martensite.

3.4.3 Deformation behavior of NiTi SMA at low temperature

Figure 10 indicates the true stress—strain curves of as-cast NiTi SMA samples under compression at various strain rates at −100 °C. Figure 11 shows the microstructures of the corresponding compressed samples. It can be obviously seen from Fig. 10 that the strain rates have little influence on the true stress—strain curves of the as-cast NiTi SMA at −100 °C. However, interestingly, the microstructure of the compressed NiTi

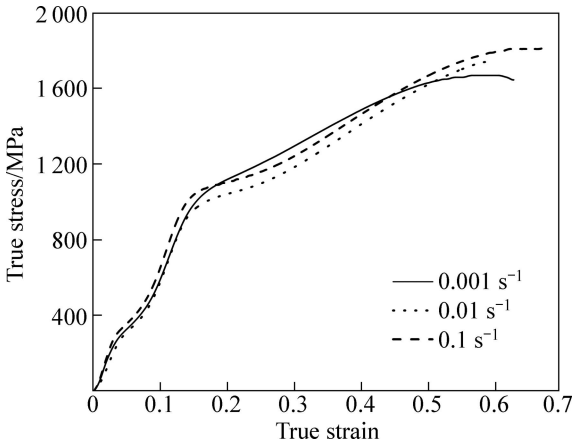

Fig. 10 True stress—strain curves of as-cast NiTi SMA at −100 °C

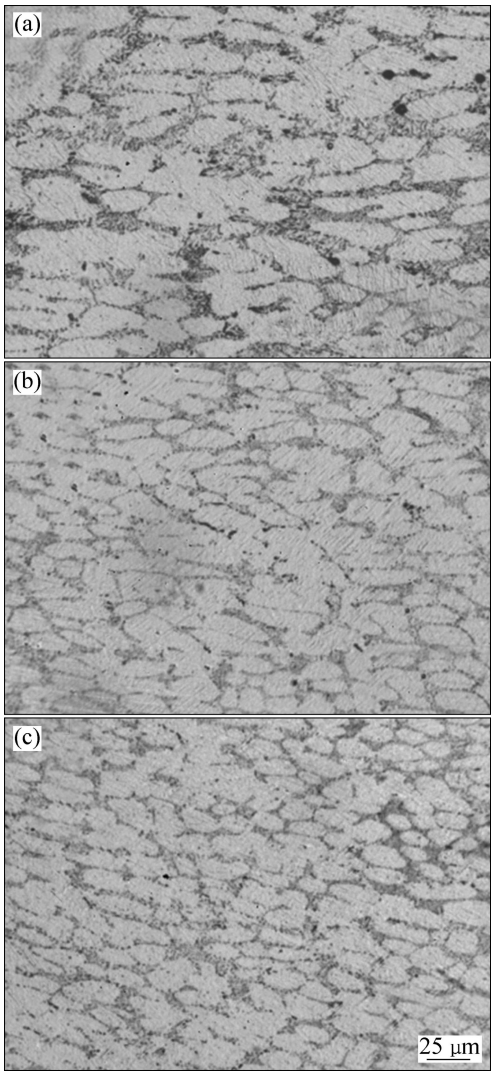

Fig. 11 OM images of compressed samples of as-cast NiTi SMA at −100 °C at different strain rates: (a) 0.1 s^{-1}; (b) 0.01 s^{-1}; (c) 0.001 s^{-1}

SMA samples at −100 °C consists of a lot of equiaxed grains instead of dendritic grains. One probable reason is that the dendritic grains of as-cast NiTi SMA are changed into equiaxed grains after plastic deformation in a martensite state at low temperature, and another probable reason is that the microstructure of the original as-cast NiTi SMA samples before compression belongs to the equiaxed grains, which are retained after plastic deformation in a martensite state at low temperature. Certainly, the two reasons need to be further validated in future work. The stress—strain curves in Fig. 10 resemble that in Fig. 8, but the plastic yielding stress of martensite at −100 °C is obviously greater than that at room temperature.

4 Conclusions

1) The microstructures of the as-cast equiatomic NiTi SMA consisted of dendritic grains and equiaxed grains are very inhomogeneous and even show severe segregation. The matrix phase of the as-cast NiTi SMA is NiTi and the Ti_2Ni phase is distributed among the NiTi matrix phases.

2) The occurrence of the Ti_2Ni phase causes the NiTi matrix to be rich in Ni, which leads to the decrease of the transformation temperatures of the as-cast NiTi SMA. The as-cast NiTi SMA consists of B19′ martensite, B2 austenite and Ti_2Ni phase simultaneously at room temperature, which is attributed to the incomplete transformation of as-cast NiTi SMA from austenite to martensite at room temperature.

3) The true stress—strain curves of NiTi SMA samples under compression at various strain rates at 750 °C indicate that NiTi SMA is sensitive to the strain rates at high temperature. At strain rates of 0.1 and 0.01 s^{-1}, the microstructure of the corresponding compressed sample simultaneously shows dynamic recrystallization characteristics because the grains are more considerably refined than those of the as-cast NiTi SMA. Under compressive deformation at strain rate of 0.001 s^{-1}, NiTi SMA shows dynamic recovery rather than dynamic recrystallization.

4) The stress—strain curves of as-cast NiTi SMA under compression at room temperature and at −100 °C are both insensitive to the strain rates, but the plastic yielding stress of martensite at −100 °C is obviously greater than that at room temperature.

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铸态镍钛形状记忆合金压缩变形行为及组织演变

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摘 要：通过真空自耗电极电弧熔炼法制备等原子比镍钛形状记忆合金铸锭。应用差示扫描量热分析、X 射线衍射分析、能谱分析和显微分析等技术研究铸态镍钛形状记忆合金在不同温度和不同应变速率压缩加载下的组织演变和变形行为。铸态镍钛形状记忆合金的显微组织由树枝晶和等轴晶组成，成分不均匀，存在偏析。在室温下，铸态镍钛形状记忆合金是 B19′马氏体、B2 奥氏体和 Ti_2Ni 的混合物。铸态镍钛形状记忆合金在高温压缩变形下对应变速率敏感，铸态镍钛形状记忆合金在 0.1 s^{-1} 和 0.01 s^{-1} 应变速率下具有动态再结晶的特征，而在 0.001 s^{-1} 应变速率下则表现出动态回复的特征。在室温和−100 ℃ 时，应变速率对铸态镍钛形状记忆合金的显微组织和真实应力应变曲线影响不大。

关键词：铸态镍钛；组织演变；变形行为；动态再结晶；形状记忆合金

(Edited by FANG Jing-hua)