Materials Research Express

PAPER

Hot deformation behavior of 60NiTi shape-memory alloy fabricated by hot isostatic pressing

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Keywords: 60NiTi shape-memory alloy, hot isostatic pressing, hot deformation behaviour, constitutive equation, finite element simulation

Abstract

The 60NiTi (Ni60wt%–Ti40wt%) intermetallic is a hard-to-process material. Understanding of hot deformation behavior is crucial for the hot working of 60NiTi. This work studied hot deformation behavior and corresponding microstructure of the hot isostatic pressed 60NiTi in the temperature range of 900 °C–1050 °C and at strain rates of 0.1, 0.01 and 0.001 s⁻¹ through a hot compression test. The flow stress and microstructure were susceptible to the hot deformation parameters. The flow stress decreased with the increase in deformation temperature and decrease in strain rate. Work hardening occurred at a small strain, then followed by softening; finally, near-dynamic equilibrium was achieved between work hardening and softening. A constitutive equation was developed to describe the effects of strain rate and temperature on flow stress. Simulation of hot deformation via the finite element method revealed the workpiece’s inhomogeneous deformation. The deformation occurred mainly in the center area of the cylindrical sample, resulting in high stress and strain concentrations in this region and causing the equiaxial grains to be compressed into prolate grains. This work can provide guidance for the hot working, such as forging and hot rolling, of 60NiTi.

1. Introduction

As a popular functional materials, near-equatomic NiTi shape-memory alloys (SMAs) have been widely used in the fields of biomedicine, sensors etc, due to their special properties, such as superelasticity, shape memory effect, and biocompatibility [1–3]. However, their engineering applications are greatly restricted by their undesirable strength and hardness [4, 5]. Ni-rich SMAs were developed by forming a hard Ni₄Ti₃ phase to solve this problem [6]. 60NiTi, which contains 60 wt.% Ni, is an alloy with high strength, hardness and low modulus. Thus, it is considered a promising material for gear, and specialized bearing applications [7–9].

In spite of the excellent properties of 60NiTi, little effort has been exerted to fabricate components out of this alloy due to the poor workability and machinability [10–12]. At present, 60NiTi components are mainly fabricated via the ingot metallurgy (IM) method [13, 14]. However, IM 60NiTi is generally unsatisfactory because of the poor mechanical properties caused by the impurities introduced during the IM process, and the inhomogeneities and segregations in the solidification microstructure [15]. Hot isostatic pressing (HIP) powder metallurgy (PM) can fabricate nearly fully dense parts with minimal impurities and a homogeneous microstructure [16–18]. Therefore, this technique has great potential for fabricating hard-to-process 60NiTi alloys [19].

The workability of 60NiTi at high temperatures is unfavorable because of the formation of abundant intermetallics, such as Ni₅Ti₃, Ni₅Ti₁₃, and Ti₂Ni phases [20, 21]. Therefore, understanding of hot deformation behavior is crucial for microstructural control and further manufacturing processes, e.g. forging, extrusion, and hot rolling. Dehghani et al investigated the hot compression deformation behavior of IM 60NiTi alloy at 950 °C–1050 °C by using experimental and computational methods [20]. Shu et al studied the hot tensile deformation of

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IM 60NiTi alloy at 650 °C–850 °C [22]. Although a high deformation temperature and a low strain rate can decrease the peak stress and steady-state stress, IM 60NiTi alloy still exhibits poor hot workability in tensile and compression deformation processes. To date, minimal attention has been devoted to the deformation behaviors at elevated temperatures and microstructural evolution of PM 60NiTi alloy, which deserve a systematical study to further utilize PM 60NiTi alloy.

To understand the hot deformation behavior and related microstructure evolution of PM 60NiTi alloy, this work investigated deformation behaviors of the 60NiTi alloy at temperatures of 900 °C–1050 °C and strain rates of 0.1, 0.01, and 0.001 s⁻¹. The flow behavior of PM 60NiTi alloy was studied, and a constitutive equation was developed to clarify the influence of hot deformation parameters on flow stress state of PM 60NiTi. Then, the evolutions of the microstructure and deformation stress and strain were characterized using experimental and simulation methods. This work contributes to the understating of the deformation behavior of PM 60NiTi alloy and provides guidance for further utilization of PM 60NiTi alloy.

2. Materials and methods

2.1. Experimental procedures

The pre-alloyed 60NiTi powder was obtained via a plasma rotating electrode technique. Table 1 listed the chemical constitution of pre-alloyed powder. The particle diameter of pre-alloyed powder ranged from 49.2 μm to 60NiTi alloy prepared by HIP was approximately 6.69 g cm⁻³, close to the theoretical density of 60NiTi (6.7 g cm⁻³), revealing low porosity of HIP-fabricated 60NiTi alloy.

|         | Ti       | Ni       | O (ppm) | N (ppm) |
|---------|----------|----------|---------|---------|
| Bal.    | 59.9     | 393      | 40      |         |

The cylindrical samples (ϕ9 × 12 mm) prepared for the hot compression test were cut from the HIP-fabricated 60NiTi through wire electrical discharge machine. The cylindrical sample’s surfaces were polished and coated with a high-temperature lubricant. The isothermal compression test is performed using a Gleeble-3800 thermal/force simulator at a temperature in the range of 900 °C–1050 °C, with an interval of 50 °C, and a strain rate of 0.1, 0.01, and 0.001 s⁻¹. The heating rate for the hot compression test is 5 °C/min. After reaching the deformation temperature, samples were kept for 300 s to homogenize the temperature before deformation. To reserve the deformed microstructures, the hot-deformed samples were water-quenched immediately. The microstructure and phase constitution of undeformed 60NiTi were systematically determined by scanning electron microscopy (SEM, backscattered electron mode, BSE; 20 kV, 3 nA), electron backscatter diffraction (EBSD; 20 kV, 10 nA), and X-ray diffraction (XRD, step size of 0.02, Cu Kα radiation). After hot deformation test, SEM and EBSD experiments were further conducted to illustrate the effects of deformation parameters on the microstructure and grain features of 60NiTi alloy. The samples prepared for EBSD experiments were first polished with polished with a 0.3 μm diamond polishing solution. Then, the surface residual stress was removed by using the Buehler VibroMet®2 Vibratory Polisher.

2.2. Finite element method

2.2.1. Geometric model and meshing method

The hot deformation behavior of 60NiTi alloy was further studied based on the finite element method (FEM), using Design of Environment for Forming (Deform-2D/3D v11.0) software. The properties of 60NiTi used in the simulation are available in [23]. The geometric dimensions of the cylindrical workpiece and dies in this simulation were ϕ9 × 12 mm and ϕ250 × 150 mm, respectively. The absolute method was used to divide the workpiece into a tetrahedral mesh with positive control of volume loss to guarantee the calculation efficiency and simulation accuracy. The minimum element size and size ratio were 1.5 mm and 2, respectively. The weighting factors of mesh distribution were 0.3 in temperature, 0.5 in surface curvature, 0.6 in strain rate, and 0.55 in strain. When the meshes were distorted and the nodes were entangled, the Deform software was utilized to conduct automatic remeshing and pass the solution information from the old meshes to the new ones [24].

2.2.2. Boundary condition

Considering the isothermal conditions, the temperatures of the bottom and top dies and the workpieces are kept the same and unchanged throughout deformation. Meanwhile, the bottom and top dies were regarded as rigid...
bodies, wherein deformation does not occur during FEM simulation. During the isothermal hot deformation of 60NiTi alloy, the upper die moved at a fixed strain rate, and the lower die was stationary.

The Deform-2D/3D software has three types of friction models, namely, Coulomb, shear, and hybrid friction. Shear friction is mostly applied in the deformation process of bulk materials, because of their shear deformation behaviors. The typical friction values vary from 0.2 to 0.9. Since the graphite lubrication is widely utilized between the mold and the workpieces, thus the friction coefficient is assumed to be 0.3 to represent this situation [25].

3. Results and discussion

3.1. Initial microstructure

Figure 1 shows initial microstructure of HIP-fabricated 60NiTi. As presented in the BSE image in figure 1(a), abundant intermetallic phases were formed within the grain interior and boundary. According to the BSE contrast and Zhou’s work [26], the bright precipitates along the grain boundaries and inside the grains are Ni3Ti phase. The black precipitates along the grain boundaries and the grey precipitates inside the grains are Ti2Ni and Ni3Ti2 phases, respectively. XRD spectrum shown in figure 1(c) further confirms that the 60NiTi prepared by HIP mainly consists of B2 NiTi, Ni3Ti and Ni3Ti2 phases. The EBSD orientation map in figure 1(d) shows an equiaxial and homogeneous microstructure, indicating the uniform properties of 60NiTi prepared by HIP. The phase map in figure 1(e) confirms the phase constitutions and contents. The fractions of Ni3Ti, Ni3Ti2, and Ti2Ni phases were 14.5%, 2.0%, and 0.5%, respectively.

3.2. Flow behavior

Figure 2 shows compressive true stress–strain curves of 60NiTi alloy at deformation temperatures of 900 °C–1050 °C and strain rates of 0.1, 0.01, and 0.001 s⁻¹. The flow stresses decreased with increasing deformation temperatures and decreasing strain rates, indicating that the flow stresses were susceptible to strain rate, and deformation temperature. This phenomenon is ascribed to low critical slip shear stress and high average kinetic energy of atoms at elevated deformation temperatures [27].

Strain rate affects both the flow stress and the deformation behavior. The true stress–strain curves at strain rates of 0.01 and 0.1 s⁻¹ in this work could be roughly divided into three stages. The first one was the work-hardening stage before reaching the peak stress, which may be attributed to the increase in dislocation density. At the second stage, the flow stress decreased significantly with the increase of strain, which may be ascribed to dynamic softening, e.g. dynamic recrystallization (DRX) and dynamic recovery (DRV) [20]. Lastly, a near-steady-state flow stage occurred when the strain was further increased. The near-dynamic equilibrium at this stage can be attributed to the balance between dynamic softening and work hardening. Under a low strain rate, such as <0.001 s⁻¹, work hardening and dynamic softening can quickly enter a near-balanced state. Therefore,
the decrease in flow stress (i.e., softening) in this study was hardly observed in the true stress–strain curves at a strain rate of 0.001 s$^{-1}$. However, under high strain rate, such as $<0.1$ s$^{-1}$, the work hardening effect may be much larger than the softening effect during hot compression tests, causing an overshoot in stress. With the increase of deformation strain, the work hardening and softening were coordinated with each other and finally reached a near-dynamic equilibrium state. Therefore, the flow stress shows a fluctuation at the beginning of the plastic deformation under high strain rate.

### 3.3. Constitutive equations

Constitutive equations can be used to describe the inherent correlation between the dynamic properties of the material and different parameters of hot deformation, e.g. strain rate, flow stress, and deformation temperature \[28\]. Therefore, studying the hot deformation behavior of HIP-fabricated 60NiTi by using constitutive equations is necessary. The classical hyperbolic function (equation \(1\)) developed by Sellars and Tegart is extensively used to clarify the relationships among the deformation temperature, strain rate, and flow stress of materials \[29\].

\[
\dot{\varepsilon} = A \exp\left(-\frac{Q}{RT}\right)[\sinh(\alpha \sigma)]^{n}
\]  

where $\dot{\varepsilon}$ is strain rate (s$^{-1}$); $Q$ is the activation energy for hot deformation (J·mol$^{-1}$); $R$ is the gas constant (8.314 J·mol$^{-1}$·K$^{-1}$); $T$ is the deformation temperature (K); $\sigma$ is the flow stress (MPa); and $A$, $\alpha$, and $n$ are material constants. $\alpha$ can be calculated with equation \(2\).

\[
\alpha = \left(\frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sigma}\right) \left(\frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sigma}\right)
\]  

The natural logarithm form of equation \(1\) is presented to be:

\[
\ln \dot{\varepsilon} = \ln A - \frac{Q}{RT} + n \ln [\sinh (\alpha \sigma)]
\]  

The linear relationship between $1/T$ and $\ln[\sinh(\alpha \sigma)]$ was obtained by taking the partial derivative of equation \(3\) to strain rate ($\dot{\varepsilon}$) when the deformation temperature is fixed.

**Figure 2.** Typical compressive true stress–strain curves of the HIP-fabricated 60NiTi at different strain rates with deformation temperatures of (a) 900 °C, (b) 950 °C, (c) 1000 °C, and (d) 1050 °C.
Under fixed strain rate, the activation energy (Q) of hot deformation was obtained by taking the partial derivative of equation (3) to 1/T as follows:

\[
\frac{1}{n} = \left[ \frac{\partial (\sinh(\alpha\sigma))}{\partial \ln \dot{\varepsilon}} \right]_T
\]

(4)

Figure 3 depicts the linear relationships between the hot deformation parameters. The average values of \(\alpha\), \(n\), \(Q\), and \(A\) were obtained by linearly fitting the lines in figure 3, i.e., \(\alpha = 0.0202\), \(n = 3.05\), \(Q = 18811.5\) Jmol\(^{-1}\), and \(A = 595341.76\). Therefore, the constitutive equation of the HIP-fabricated 60NiTi was expressed as:

\[
\dot{\varepsilon} = 595341.76 \exp \left( -\frac{18811.5}{RT} \right) [\sinh(0.0202\sigma)]^{1.05}
\]

(6)

The Zener–Hollomon parameter (Z) is often utilized to investigate the relationships between the changes in deformation temperature or strain rate and the stress–strain behavior [20, 30, 31]. The expression for hot deformation behavior of the material represented by Z is illustrated in equation (7):

\[
Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right)
\]

(7)

The relationship between flow stress (\(\ln[\sinh(\alpha\sigma)]\)) and \(\ln Z\) was obtained by taking the natural logarithm of equation (7):

\[
\ln Z = \ln A + n \ln [\sinh(\alpha\sigma)]
\]

(8)

Figure 4 shows the calculated values of \(\ln Z\) under different hot deformation parameters and the linear fitting curve between \(\ln Z\) and \(\ln[\sinh(\alpha\sigma)]\). The adjusted R-square of the fitting curves is 0.9563, indicating that the fitting curve shows an excellent linear correlation between \(\ln[\sinh(\alpha\sigma)]\) and \(\ln Z\). This result also demonstrates that the HIP-fabricated 60NiTi alloy satisfied the Arrhenius relationship among deformation temperature,
strain rate, and flow stress during deformation at elevated temperatures. Therefore, the flow stress under different deformation parameters can be effectively expressed by the Zener–Hollomon parameter.

3.4. Simulation of deformation behavior
The constitutive relations between stress and strain were used to investigate the hot deformation behavior via the Deform-2D/3D software. Taking the sample deformed at 900 °C and at a strain rate of 0.001 s⁻¹ as an example, the deformation process was simulated, and the results are shown in figure 5. Deformation first occurred in the center area of the cylindrical sample, resulting in stress and strain concentrations in this region. The effective strain and stress gradually increased with the increase of height reduction. The maximum effective strain and stress were observed in the cylindrical center and edge areas of the top and bottom surfaces. The minimum effective strain and stress were located in the cylindrical edge of the center and the center regions of the bottom and top surfaces. The variation in the average effective stress and strain with the increase in height reduction was consistent with the true stress–strain curves (figure 2). These simulation results on deformation behavior can provide guidance for the hot working, such as forging and hot rolling, of 60NiTi alloy. The inhomogeneous distribution of strain and stress can also explain the microstructure variation in different regions of the deformed sample.

3.5. Deformed microstructure
BSE images shown in figure 6 illustrate the microstructural variations in the center and edge areas of the HIP-fabricated 60NiTi alloy after deformation at 900 °C and different strain rates. Compared with microstructure of undeformed 60NiTi (figure 1), the microstructure of 60NiTi after hot deformation occurred obvious changes.
On the one hand, the equiaxial grains were prolonged in the direction perpendicular to the load, and the distortion of the grains in the center area was much larger than that in the edge area. The difference in grain distortion can be explained by the simulation results of stress and strain shown in figure 5. During hot deformation, stress and strain were mainly concentrated in the cylindrical center, resulting in serious distortion of the grains in this region. On the other hand, after hot deformation at 900 °C, the Ni$_3$Ti$_2$ phase could not be observed in the BSE image; it may have been transformed into the stable Ni$_3$Ti phase at a high temperature [26]. Meanwhile, the Ni$_3$Ti phase was refined by the serious deformation at 900 °C, and most of the Ni$_3$Ti laths were arranged along the direction perpendicular to the load.

The effects of deformation temperatures and strain rates on the deformed microstructure in the center area of the 60NiTi alloy are illustrated in figure 7. The deformation temperatures had obvious effects on the microstructural features, whereas the strain rates affected the deformed microstructure only slightly. The distortion degree of the grains was kept almost consistent under different strain rates and deformation temperatures. The Ni$_3$Ti phase was gradually dissolved into the B2 NiTi matrix with the increase in deformation temperatures from 900 °C to 1050 °C; this result is consistent with Zhou’s study on the heat treatment of 60NiTi [26].

EBSD experiments were conducted on the central area of the samples deformed at a strain rate of 0.001 s$^{-1}$ to further study the grain structures after hot deformation. As shown in figure 8, all the equiaxial B2 NiTi grains were compressed into prolate grains. Meanwhile, Ni$_3$Ti precipitates were re-arranged along the direction perpendicular to the load for the samples deformed at 900 °C. With the increase of deformation temperature, the Ni$_3$Ti precipitates were gradually dissolved into the B2 NiTi matrix. These microstructural variations observed by EBSD are consistent with the BSE images presented above.

Figure 6. SEM-BSE images showing the microstructure in the center and edge areas of the HIP-fabricated 60NiTi alloy after deformation at 900 °C and different strain rates: (a), (c), and (e) show microstructure of the edge area after deformation at 900 °C at strain rates of 0.1, 0.01, and 0.001 s$^{-1}$, respectively; (b), (d), and (f) show the microstructure of the center area after deformation at 900 °C at strain rates of 0.1, 0.01, and 0.001 s$^{-1}$, respectively.
Figure 7. SEM-BSE images showing the microstructure in the center area of the HIP-fabricated 60NiTi alloy deformed under different strain rates with deformation temperatures of (a)–(c) 950 °C, (d)–(f) 1000 °C, and (g)–(i) 1050 °C.

Figure 8. EBSD images showing the grain structures of the samples deformed at a strain rate of 0.001 s$^{-1}$ and deformation temperatures of: (a) 900 °C, (b) 950 °C, (c) 1000 °C, (d) 1050 °C.
4. Conclusions

The hot deformation behavior of HIP-fabricated 60NiTi was investigated in the temperature range of 900°C–1050°C and at strain rates of 0.1, 0.01, and 0.001 s⁻¹ by using constitutive equations and FEM. The variations in the microstructure after hot deformation were also examined. The main results and conclusions are summarized as follows:

1. The flow stress of the HIP-fabricated 60NiTi alloy was susceptible to the deformation temperatures and strain rates. The flow stress decreased with the increase in deformation temperature and decrease in strain rate. Work hardening occurred at a small strain, then followed by softening. Finally, near-dynamic equilibrium was achieved between work hardening and softening.

2. Constitutive equations were developed using true stress–strain data. The deformation activation energy was calculated to be 188.111 kJ/mol. The constitutive equation for the HIP-fabricated 60NiTi can be expressed as: 
   \[ \dot{\varepsilon} = 595341.76 \exp \left(-\frac{18811.5}{RT}\right)\sinh(0.0202\sigma) \] 
   The adjusted R-square of the fitting curves (lnZ to ln [\sinh(\alpha\sigma)]) was 0.9563, indicating that the flow stress under different deformation parameters can be effectively expressed by the Zener–Hollomon parameter.

3. The simulation of hot deformation via FEM revealed the workpiece’s inhomogeneous deformation. Deformation mainly occurred in the center area of the cylindrical sample, resulting in stress and strain concentrations in this region.

4. The initial microstructure of the HIP-fabricated 60NiTi showed equiaxial grains within coarse precipitations. After hot deformation, the equiaxial grains were compressed into prolate grains, and the distortion of the grains in the center area was much larger than that in the edge area, which can be explained by the inhomogeneous stress and strain distribution. With the increase of deformation temperatures, the Ni₃Ti precipitates are gradually dissolved into the B2 NiTi matrix.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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