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Time-temperature-transformation diagram for the martensitic transformation in a titanium-nickel shape memory alloy

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ABSTRACT

We have constructed a time-temperature-transformation (TTT) diagram for the martensitic transformation in a Ti-51.3Ni (at%) alloy. The transformation proceeds when the specimen is held at fixed temperatures between 110 K and 140 K, but not at other temperatures. The time required for the formation of 0.1% of the martensite phase is plotted as a TTT diagram. It exhibits a C-curve with a nose temperature near 130 K. Application of a thermal activation model to the TTT diagram suggests that the size of a nucleus of the martensite phase is about $(2.2\ \mathrm{nm})^3$.

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1. Introduction

Martensitic transformations are traditionally classified into two groups from the view point of kinetics: athermal and isothermal [1]. It is considered by many researchers that the volume fraction of the martensite does not depend on time in an athermal transformation, but does so in an isothermal transformation. However, we consider that all martensitic transformations are intrinsically isothermal, and an athermal transformation is a special case of isothermal transformations as pointed out by Kurdjumov [2]. This interpretation has been supported by several experimental results. One example is that an iron based alloy exhibiting a clear C-curve in its time-temperature-transformation (TTT) diagram transforms instantaneously when a pulsed magnetic field is applied [3]. Another example is that an iron based alloy which has a clear M_s (transformation start temperature) exhibits a C-curve in its TTT diagram when a hydrostatic pressure is applied [4]. These results imply that athermal and isothermal transformations are intrinsically the same in nature.

Since then, the isothermal nature of martensitic transformation has been observed in many alloys that are believed to show athermal transformations. Examples are martensitic transformations in Fe-Ni [4], Cu-Al-Ni [5,6], and Ni-Mn-In Refs. [7–9] alloys. The isothermal nature was also reported in the B2-B19′ transformation of binary Ti-Ni shape memory alloys. Kustov et al. [10] observed that in a Ti-50.2Ni (at.%) alloy the B2-B19′ transformation proceeds when held between M_s and M_f (transformation finish temperature). After that, our group observed in a Ti-51.2Ni (at.%) alloy that the B2-B19′ transformation occurs while the specimen is held above its M_s temperature [11] although Otsuka et al. [12] reported that a Ti-50Ni(at.%) alloy does not show an isothermal transformation when held above its M_s. However, the TTT diagram for the B2-B19′ transformation has not been constructed yet, in spite of the fact that the TTT diagram is fundamental for understanding the isothermal nature of the transformation. The aim of this study is to construct a TTT diagram for the B2-B19′ transformation in a binary Ti-Ni shape memory alloy. In order to construct a TTT diagram, it is desirable to suppress the appearance of the M_s temperature during the cooling process. If M_s exists, it is practically impossible to draw a curve corresponding to a constant fraction of transformed product in the TTT diagram. In this study, we used a Ti-51.3Ni (at.%) alloy for the construction of the TTT diagram because M_s is absent in this alloy.

2. Experimental procedure

A button ingot of Ti-51.3Ni (at.%) alloy was prepared by arc

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melting. It was homogenized at 1273 K for 86.4 ks in evacuated quartz tubes and then quenched in ice water. A specimen for electrical resistivity measurements (10 mm × 2 mm × 0.3 mm) was cut from the ingot and the surface was polished using emery paper. It was then heat-treated at 1273 K for 3.6 ks followed by quenching in ice water. The surface of the specimen was polished using an electrolyte composed of 10% perchloric acid and 90% acetic acid. Four gold wires were attached to the specimen, and the specimen was mounted on a stage using silicone grease. Electrical resistivity of the specimen was measured by an alternating current four terminal method with a current of 100 mA and a frequency of 10 Hz.

3. Results

Fig. 1 shows the electrical resistivity curve of the Ti-51.3Ni (at.%) alloy. The measurement was made during the cooling process and the subsequent heating process with a fixed cooling and heating rate of 2 K/min. The resistivity increases with decreasing temperature. There is no detectable hysteresis between the cooling and heating processes. The resistivity curve resembles that of Ti-Ni alloys containing 51.5 at.% or more of Ni [13], and it has been confirmed previously that the Ti-51.5Ni (at.%) alloy does not show a thermal-induced B2-B19′ transformation [13,14]. Therefore, from the resemblance of resistivity curve, we may interpret that the Ti-51.3Ni (at.%) alloy does not show an apparent B2-B19′ transformation in the continuous cooling process.

In order to detect the martensitic transformation by holding at a fixed temperature, we measured the electrical resistivity through the holding process. Before holding, the specimen was initially cooled from 300 K to the target temperature at a cooling rate of 2 K/min. The fluctuation of the temperature of the stage during the holding process was within 0.01 K Fig. 2(a) shows the change in resistivity when the specimen was held at the temperatures indicated in the figure. The scale of the vertical axis is nearly the same as that in Fig. 1. At this scale, the change in resistivity is difficult to observe. However, if we expand the vertical axis, we notice a slight change in resistivity.

Fig. 2(b) is a magnified resistivity curve at 130 K. At this temperature, the resistivity decreases as soon as the holding is started. The decrease in resistivity implies progress of the B2-B19′ transformation because the electrical resistivity of the B19′-phase is lower than that of the B2-phase. We may neglect the possibility of the B2-R or the B2-B19 transformation because the resistivity should increase instead of decrease associated with these

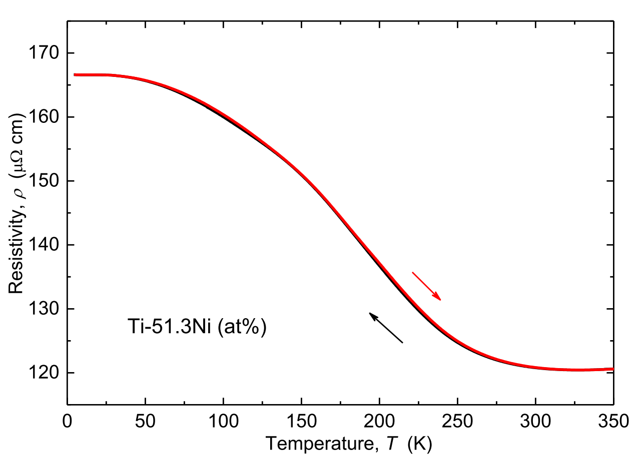

Fig. 1. Temperature dependence of electrical resistivity of the Ti-51.3Ni (at.%) alloy measured in the cooling and subsequent heating process with a cooling and heating rate of 2 K/min.

transformations. In addition we may neglect the influence of diffusion of atoms at the holding temperature. A similar decrease in resistivity was observed at 140 K, 120 K and 110 K. Therefore, we may conclude that the isothermal B2-B19′ transformation occurs at 110 K, 120 K, 130 K and 140 K.

Fig. 2(c) shows the magnified resistivity vs temperature curves at 100 K. At this temperature, the resistivity is nearly independent of the holding time. Similar results were obtained at 80 K, 90 K, 150 K, 160 K and 170 K. Therefore, we may conclude that the specimen does not show martensitic transformation in the holding process up to 2.7 ks at these temperatures. Incidentally, we notice a slight increase in resistivity in the time interval of between 0 and 0.5 ks The reason of this slight increase is probably due to a slight decrease in temperature of the specimen during the initial holding process. According to a recent holding experiment made by Ji et al. [15] using a Ni-rich Ti-Ni alloy (Ti-51.3Ni (at.%) according to their report), the R-phase forms in the alloy when held at a fixed temperature. However, the large increase in resistivity reported by Ji (nearly 1%) was not detected in the present study. The increase in resistivity is only 0.02% in Fig. 2(c). Therefore we may regard that the R-phase is not formed in the present specimen although the nominal composition of the alloy is the same. There is one more difference between the present result and that reported by Ji et al.; the M_s temperature is missing in the present specimen, but it is near 178.6 K in the specimen reported by Ji et al. The reason of these differences is not clear, but could be caused by a slight difference in composition or heat-treatment.

In order to construct a TTT diagram for the B2-B19′ transformation, we roughly evaluate the time ($t_{0.1}$) necessary for the formation of 0.1% of the B19′-phase during the holding process. For the evaluation of $t_{0.1}$, we make two assumptions. First, we assume that the resistivity of the B19′-phase is smaller than that of the B2-phase by 20% based on a previous study of electrical resistivity in a Ti-51.2Ni (at.%) alloy [12]. Second, we assume that the fraction of the B19′-phase is proportional to the change in resistivity. Under these two assumptions, formation of 0.1% of the martensite phase corresponds to a 0.02% decrease in resistivity. For example, $t_{0.1}$ is 0.18 ks at 130 K as indicated in Fig. 2(b). The time $t_{0.1}$ thus evaluated at other temperatures is plotted as a function of the holding temperature in Fig. 3 by solid circles. We can see that the TTT diagram exhibit a C-curve with a nose temperature located near 130 K. Incidentally, the open circles in Fig. 3 indicate that the decrease of 0.02% in resistivity was not observed up to the maximum holding time of 2.7 ks at 100 K and 150 K.

4. Discussion

The C-curve in the TTT diagram indicates that the B2-B19′ transformation occurs by a thermal activation process. One of the authors has proposed a thermal activation model for martensitic transformations to evaluate the size of a nucleus of the martensite phase [16], and it was applied for isothermal transformations in iron-based alloys [16,17] and in a Ni-Co-Mn-In alloy [18]. In the following, to evaluate the size of the nucleus, we apply the model to the B2-B19′ transformation in the Ti-51.3Ni (at.%) shape memory alloy. In the model [16], a potential barrier Δ (activation energy per mole) is considered to exist between the parent and the martensite phases and is assumed to depend on the free energy difference between the B2- and B19′-phases Δg ($=g^{B2}$-$g^{B19'}$) as $\Delta = \delta - \Delta g$, where δ is the potential barrier per mole at the equilibrium temperature T_0 between the two phases. Then the transformation occurs by overcoming the potential barrier after a cluster composed of m^* atoms is formed. The probability P of forming such an activated cluster is given as [16].

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