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Acta Materialia 53 (2005) 4009–4018
Acta MATERIALIA
www.actamat-journals.com

Effect of pre-deformation at room temperature on shape memory properties of stainless type Fe–15Mn–5Si–9Cr–5Ni–(0.5–1.5)NbC alloys

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Received 19 April 2005; accepted 26 April 2005
Available online 11 July 2005

Abstract

Substantial improvement of shape memory properties of the stainless type Fe–15Mn–5Si–9Cr–5Ni–(0.5–1.5)NbC (mass%) alloys has been achieved by pre-deformation at room temperature. It shows that a suitable pre-extension can result in excellent shape memory effect (SME). A 90% shape recovery of an initial 4% strain and 200 MPa shape recovery stress are attained when the Fe–15Mn–5Si–9Cr–5Ni–0.53Nb–0.06C alloy is 12% pre-extended at room temperature, being followed by aging at 1070 K for 10 min. If the pre-extension is more than 12%, the shape recovery is reduced with an increasing amount of the pre- extension. It is found by X-ray diffraction and transmission electron microscopy (TEM) that such a decrease in shape recovery is due to the formation of α' phase (body-centered cubic). The mechanism to obtain a nearly perfect shape memory recovery at 12% pre-extension is studied by atomic force microscopy and TEM. It is concluded that the uniform distribution of stacking faults associated with fine NbC precipitates produces the single variant martensite plates, which is the key factor in obtaining an excellent SME.

Keywords: Fe-based shape memory alloy; Niobium carbide; Pre-extension; Atomic force microcopy; Shape recovery

1. Introduction

Since it was found that Fe–30Mn–1Si (mass%) single crystal possesses the shape memory effect (SME) [1], Fe–Mn–Si based shape memory alloys (SMAs) have been studied actively over the past 20 years due to their obvious technological advantages, such as low cost, good mechanical properties and wide transformation hysteresis [2–8]. Enormous efforts have been paid to clarifying the composition dependence of the shape memory recovery, shape recovery stress and corrosion resistance in the Fe–Mn–Si SMAs with addition of Cr, Ni and Co, etc., with the aim of developing new shape memory materials to substitute for the expensive NiTi-based SMAs. Studies of physical and mechanical properties of these alloys together with microstructural observation of the γ(face-centered cubic (fcc)) $\leftrightarrow$ ε(hexagonal close-packed (hcp)) transformation have been also performed. Hitherto, two typical groups of Fe–Mn–Si SMAs, namely, Fe–28Mn–6Si–5Cr with a fairly good SME but poor corrosion resistance and Fe–14Mn–5Si–9Cr–5Ni with good corrosion resistance but poor SME, have been developed, but there is still no substantial industrial application apart from several trials for joining pipes in practical engineering [9]. One of the main reasons is that, in order to improve the shape recovery to an extent of 80–90%, a thermo-mechanical treatment, the so-called ‘‘training’’, must be carried out. Since this treatment consists of several cycles of deformation and heat treatment, it not only makes the manufacturing

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1359-6454/$30.00
doi:10.1016/j.actamat.2005.04.035

process more complex, but also increases the production cost of the SMA materials. Furthermore, it is impossible to apply such a treatment to the materials with somewhat complex shapes. Obviously, such a shortcoming has to be overcome for practical application.

The SME of Fe–Mn–Si-based SMAs is related to the $\gamma \rightarrow \varepsilon$ transformation induced by external stress and subsequent reversion of $\varepsilon \rightarrow \gamma$ on heating [2–4]. It was thought that a high critical stress for unrecoverable slip deformation and low critical stress for stress-induced martensite formation are necessary for good SME [5]. On the basis of this principle, some researchers have tried to strengthen the fcc matrix by strain hardening, and some have added C, N to produce the second phase to strengthen austenite, with the aim of avoiding the complicated process of "training", but no remarkable improvement in shape memory properties was achieved without performing the "training" process [10,11].

Observation on the microstructures in the "trained" samples has been actively carried out on different scales from micrometer to sub-nanometer by Kajiwara and co-workers [12–17]. They have concluded from such observations that, to realize a perfect shape memory effect, the following three conditions must be fulfilled: (1) the stress-induced martensite plate must be a single variant although there are three possible variants for a {1 1 1} habit plane [12,13,17]; (2) the width of martensite plate should be extremely small [13–15]; (3) these thin martensite plates must be uniformly distributed [13].

In 1999, Kajiwara proposed [13] that small coherent precipitates in austenite would realize the latter two conditions. He and his co-workers later succeeded in producing a remarkable improvement of the shape memory properties of conventional alloys without the "training". The key points for such an epochal improvement are as follows: (1) to add a small amount of Nb and C elements to the conventional alloys, (2) to carry out aging treatment at an appropriate temperature to generate very small particles of NbC carbides. It is thought that these NbC precipitates not only provide nucleation sites for stress-induced transformation ($\gamma \rightarrow \varepsilon$), but also increase the strength of the austenite. Following this proposal, two groups of alloys involving Nb and C, namely, Fe–28Mn–6Si–5Cr–(0.5–1.5)NbC and Fe–15Mn–5Si–9Cr–5Ni–(0.5–1.5)NbC alloys were developed. It was proved that their shape memory properties are far better than those of the conventional alloys without NbC [18,19]. More recently, they reported that shape memory properties of Fe–Mn–Si-based SMAs containing Nb and C could be further improved by pre-rolling at 870 K or at room temperature, followed by aging. The effects of aging time, aging temperature and pre-rolling at elevated temperature and room temperature on shape memory properties have been extensively investigated and a lot of factors to improve SME have been found [20–23]. Most of the results in these investigations suggest potential application of the alloys in the future.

A simple, convenient and less-costly treatment is, of course, recommended for industry application. In this view, the pre-deformation at room temperature followed by aging will be the most appropriate thermo-mechanical treatment. In the previous work by Baruj et al., it was shown that the effect of pre-rolling at room temperature on SME is largely dependent on the amount of rolling, especially, for the Fe–15Mn–5Si–9Cr–5Ni–(0.5–1.5)-NbC alloy, i.e., the shape recovery is rapidly decreased with increasing amount of rolling although the shape recovery stress is rather increased. The conventional Fe–14Mn–5Si–9Cr–5Ni alloy was originally developed as a corrosion-resistant SMA [24] that is almost equivalent to the 304 type stainless steel. So, these kind of Fe–Mn–Si-based SMAs are often called the stainless type SMAs. In our preliminary corrosion test, the modified Fe–15Mn–5Si–9Cr–5Ni alloys containing small amounts of Nb and C were also found to be highly corrosion resistant. Therefore, for this modified 15Mn alloy containing Nb and C, it is very important to find the reason for the degradation of shape recovery in the case of large amount of pre-rolling at RT. It is expected that the findings may give important information for alloy design of corrosion resistant Fe–Mn–Si-based SMAs. In the present work, tensile deformation, not rolling, is adopted as pre-deformation because the amount of deformation can be better controlled than in rolling.

2. Experimental

2.1. Alloy and sample preparation

Three alloys, i.e., Fe–15Mn–5Si–9Cr–5Ni–0.53Nb–0.06C, Fe–15Mn–5Si–9Cr–5Ni–1.06Nb–0.12C and Fe–15Mn–5Si–9Cr–5Ni–1.59Nb–0.18C (mass%), were prepared by vacuum induction melting. After being forged and hot-rolled, the alloys covered by a SiO_2 film were heat-treated at 1470 K for 10 h for homogenization. The extension samples of with 0.7 mm thickness and 1 mm width with the gauge length of 15 mm were cut from the solution treated materials. These samples were extended by various amounts at room temperature with a mechanical testing instrument and then subjected to aging at 1070 K for 10 min in evacuated quartz capsules to produce NbC precipitates (On heating to 1070 K, the stress-induced martensite generated by the room temperature extension are reverse-transformed to austenite.) After aging, these samples were then quenched into water and chemically polished to remove the surface layer. By aging, the three alloys listed above are supposed to contain 0.5, 1.0 and 1.5%NbC (mass%). Hereafter, we call these alloys 15Mn0.5NbC, 15Mn1.0NbC and 15Mn1.5NbC, respectively.