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Shape Memory Properties and Related Transformation Behavior in a Pre-deformed Sample at Room Temperature in Fe-15Mn-5Si-9Cr-5Ni-0.5NbC alloy

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Since Kajiwara and his coworkers found that the shape memory effect (SME) in Fe-Mn-Si based shape memory alloy (SMA) can be greatly improved by producing fine NbC precipitates, new SMAs without training have been developed. It was previously reported that SME of the Fe-Mn-Si based alloys containing Nb and C is further improved by pre-rolling of austenite at 870K or at room temperature before aging. In the present work, the effect of the pre-tensile deformation, in stead of rolling, at room temperature on shape memory properties and related transformation behavior is investigated. The new finding is that elongation at room temperature, followed by aging at 1070K for 10min, can substantially improve the SME. A shape memory recovery of 90% is obtained for 4% initial strain in Fe-15Mn-5Si-9Cr-5Ni-0.5NbC (mass %) alloy if the alloy is pre-elongated by 12%. This effect on SME is comparable to the case of the pre-rolling of austenite at 870K. A D-3000 atomic force microscopy was employed to observe the martensitic transformation in the samples with such treatment and the mechanism of the influence on SME is discussed.

Keywords : iron based shape memory alloy, shape recovery, shape recovery stress, niobium carbide, pre-tensile deformation

1. Introduction

Recently Kajiwara and his co-workers found that a small amount of Nb and C addition in Fe-Mn-Si based shape memory alloys could substantially improve the shape memory effect (SME) [1, 2]. The key point for such an improvement is to produce very fine NbC precipitates in the austenite by aging treatment. Very recently, further improvement on shape memory recoveries has been obtained in these NbC-containing Fe-Mn-Si based alloys. For example, 90% of an initial strain of 4% is recovered [3, 4] if an Fe-15Mn-5Si-9Cr-5Ni-0.5NbC alloy in austenitic state is pre-rolled by 10%-30% at 870K followed by aging at 1070K for 10min. This is remarkable improvement compared to only 65 % shape memory recovery for the non pre-rolled alloy.

In the present study, the effect of pre-tensile deformation at room temperature on SME has been investigated for Fe-15Mn-5Si-9Cr-5Ni-0.53Nb-0.06C alloy (mass %), which has fairly good corrosion-resistance property. The amount of deformation can be more easily controlled for tensile deformation than for rolling and, of course, the deformation at room temperature is more attractive for actual industry application.

2. Experimental

The alloy was prepared by vacuum induction melting. An ingot of 45×64×100mm^3 was forged and hot-rolled to 20mm thickness. After homogenization treatment at 1470K for 10h, the tensile samples with 0.7mm thickness and 1mm width with the gauge length of 15mm were prepared by spark cutting. Most of the samples were pre-extended by various amounts at room temperature in a mechanical testing instrument, and then heated at 1073K for 10min in evacuated quartz capsules. This heat treatment has two purposes: one is to reverse-transform the martensite formed during pre-extension at room temperature and the other is to produce NbC precipitates. These samples were quenched into water and chemically polished, and then all of the samples were initially strained about 4% by extension. The shape memory recovery was measured by observing the length change after the samples were heated to 870K. To measure the recovery stress, the 4% strained samples were fixed in a mechanical testing instrument, heated up to 670K and cooled down to room temperature, in which the shape recovery stresses were recorded automatically. A D-3000 atom force microscopy (AFM) was employed under a taping mode to observe the transformation.

3. Results

3.1 Shape recovery

Fig.1 shows the shape recovery for 4% initial strain when various amounts of pre-extension were applied at room temperature, in which the effect of pre-extension is clearly seen. The shape recovery is increased with increase of the pre-extension up to 12 %, where the shape recovery achieves a peak value, then with further pre-extension, it is decreased rapidly with increasing amount of pre-extension. At 12% pre-extension, 90% shape recovery is obtained, which is comparable to the result of the same alloy pre-rolled 10 % at 870 K [5, 6]. Such a good shape memory effect of the alloy obtained by pre-deformation at room temperature will be more attractive to engineering application.

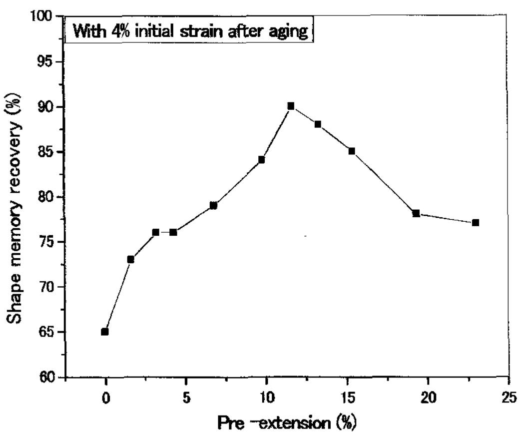

Fig.1. Relationship between the pre-deformation at room temperature and the shape recovery

3.2 Shape recovery stress

Shape recovery stress is another important parameter to evaluate Fe-based shape memory alloys, especially in the case of pipe jointing or fastening materials. The recovery stresses for different amounts of pre-extension were measured by heating-cooling cycle between room temperature and 670K, as shown in Fig.2. All the samples were given about 4 % initial strain after aging at 1070 K for 10 min before the test. It is seen that, with increase of the amount of pre-deformation at room temperature, the recovery stress is increased. Normally, the curves can be divided into four stages. On heating, the recovery stress first decreases with temperature by thermal expansion of the sample, then with further temperature increase, the ε (hcp) to γ (fcc) transformation takes place and the recovery stress begins to increase with temperature. On cooling, the recovery stress further increases due to the thermal contraction of the sample and reaches the maximum. Then, around the point where the austenite begins to re-transform into martensite, the recovery stress starts to decrease. It should be noted that, until room temperature, the shape recovery stresses of the alloy for pre-extension more than 10% are above 200MPa. The reason for such high recovery stresses will be due to the strength increase of the austenite after pre-extension and subsequent aging treatment as indicated in Fig. 3. Fig. 3 shows stress-strain curves for various amounts of the pre-extension. The deformation in these curves is, of course, mainly by stress-induced martensitic transformation, but the increase in flow stress with increasing pre-extension implies the substantial austenite strengthening.

Considering a potential application as pipe joint, the problem is that the coupling SMA material has to be partly recovered before it actually contacts the pipe. From this point of view, a simulation experiment was carried out, i.e., the tensile sample without fixing the sample heads was heated to a temperature to get a free recovery, then fixed to the mechanical testing instrument to measure the recovery stress. The recovery stress as a function of the freely recovered strain is shown in Fig. 4, in which we can see the alloy still exhibits a high recovery stress of about 170 MPa even after a free recovery of 2 %. Such a high recovery stress is enough for the fastening force in practical application.

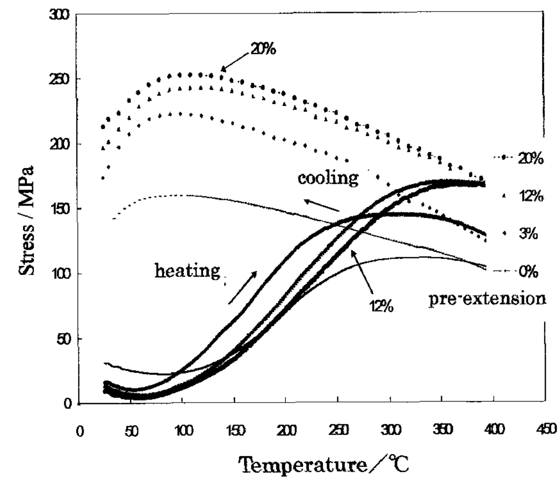

Fig.2. Shape recovery stress vs. temperature for various pre-extensions. A 4% initial strain was given after aging at 1070K for 10 min before the test.

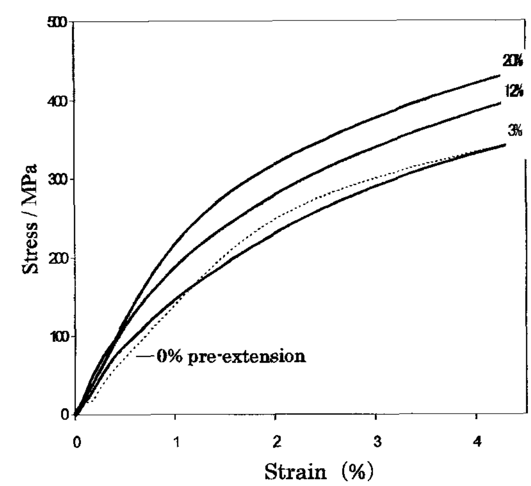

Fig.3. The stress-strain curve for various amounts of pre-extension

3.2 AFM observation

Atomic force microscopy (AFM) is a useful instrument to investigate the fcc/hcp transformation of the Fe-Mn-Si based shape memory alloys [5, 8, 9]. From the original color AFM picture, a different martensite variant can be easily distinguished.

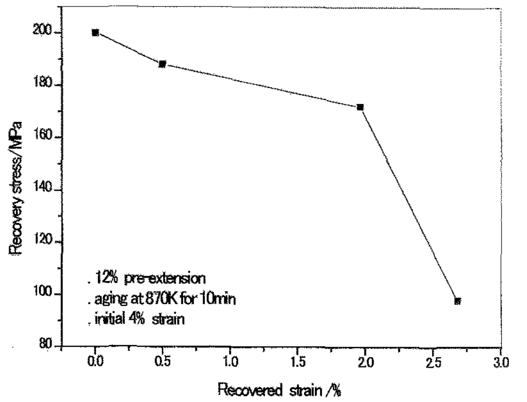

Fig.4. Relationship between the recovery stress and the recovered strain for the 12% pre-extended sample.

In this investigation, the samples with different amounts of pre-extension were aged at 1070 K for 10min, and then the samples were chemically and electrically polished to remove the surface layer and obtain a very clean surface. These samples were deformed step by step to get different amounts of deformation at room temperature and heated to different temperatures up to 670K stepwise. Fig.5 shows an example for a series of the AFM pictures taken by such stepwise deformation and heating of a 12% pre-extended sample. We can see in this figure that when the alloy deformed by 1.2 %, very fine parallel martensite plates appeared and with increasing deformation, more martensitic transformation was induced by forming many parallel martensite plates. On heating, the martensite begins to reverse-transform at 450 K. It seems that they split into thinner plates and gradually disappear on further heating. According to the previous work on the "trained sample" of the conventional Fe-Mn-Si based alloy with no NbC [8, 9], the most important requisite to achieve good SME is that the stress-induced martensite plates should have the same variant, i.e., the same color in the original AFM picture, but in the case of Fig. 5, it seems that the martensite plates are not of the same variant as inferred from the different contrast (i.e., different colors in the original AFM) of their plates. In fact, the tilt angles of martensite plates measured along the line in Fig. 5(c) revealed that the plates A (medium contrast) have a tilt angle of 3°, the plates B (weak contrast) 1.5°and the plates C (strong contrast) 8°, which means that they do not belong to the same variant. Nevertheless, these martensite plates disappeared on heating as seen in Figs. 5(d)-(f). This fact indicates that they have been reverse-transformed by taking the same atomic path as that of the forward transformation. This behavior is different from those observed in the pre-rolled sample of Fe-28Mn-6Si-5Cr-0.5NbC [3, 5]. However, in the other regions of the same sample, only the same variant of the martensite plates was observed. So we need a further investigation to draw a consistent conclusion.

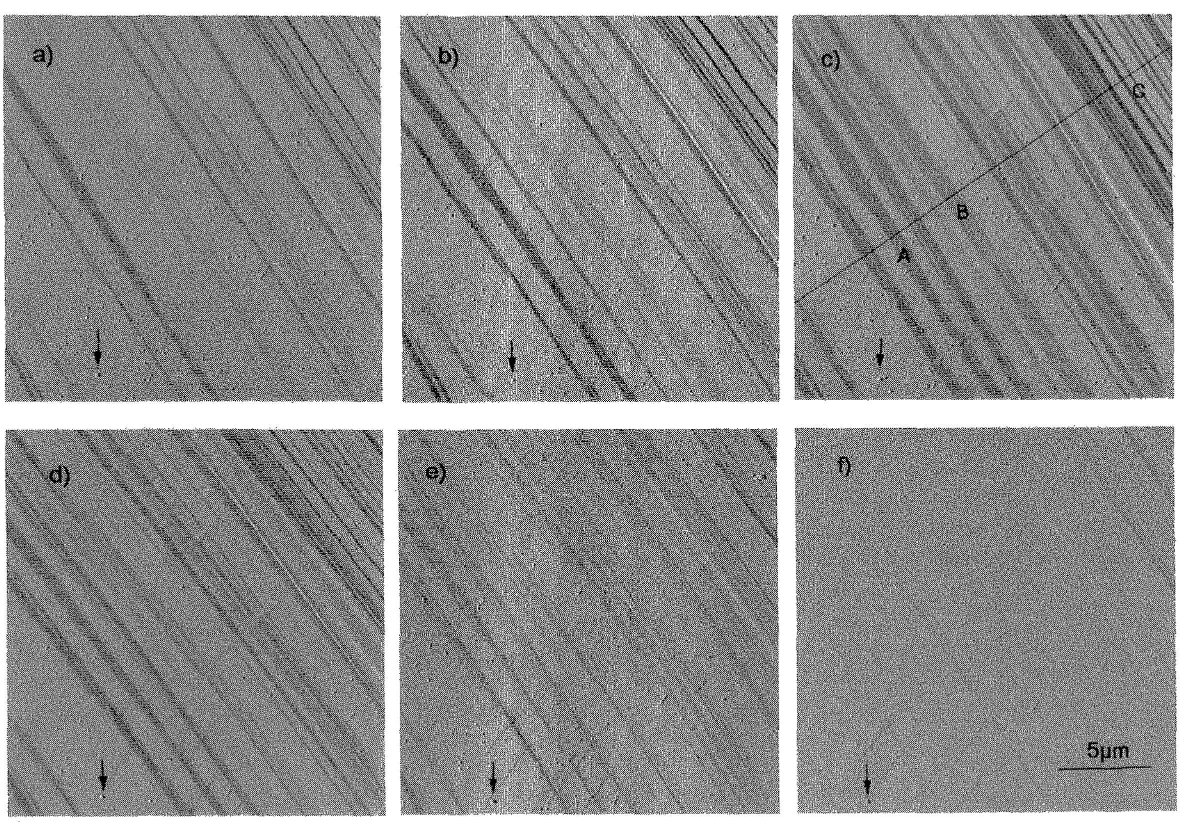

Fig 5. AFM images taken stepwise in the 12 % pre-deformed sample. a)-c): successive stage of tensile deformation, being deformed by 1.2%,2.4% and 4.0%. d)- f): shape recovery stage, 23% recovery at 450K, 70% at 500K and 90% at 670K, respectively. The small arrow in each picture indicates the identical point in the observed area.

4. Discussion

The result presented above shows that pre-extension at room temperature obviously influences the shape memory properties of Fe-15Mn-5Si-9Cr-5Ni-0.5NbC alloy. Firstly, according to a very recent report by Baruj et al. [6], the shape recovery for 4 % initial strain is only 80-60 % for 10-30 % cold rolling, while it reaches 90 % for 12 % pre-extension although rapidly decreased with increasing amount of the pre-extension. The reason for a better shape recovery in the latter case is considered due to that direction of tensile deformation for 4 % initial strain is the same as that of the pre-extension. This will make a similar effect to the "training" in the conventional alloy.

The decrease of the shape recovery in the case of pre-extension of more than 12 % will be attributed to the formation of bcc phase, which could be proved by a simple test with use of a magnet; that is, the sample was strongly attracted by the magnet if extended more than 12 %.

As for the shape recovery stress, the effect of pre-deformation is nearly the same for either the cold rolling or tensile deformation. A recent study by Baruj et al. [6] showed that the recovery stresses are 230-300 MPa for zero recovered strain and 150-180 MPa for 2 % recovered strain, which are comparable to the present results.

5. Summary

1) The shape memory properties of the Fe-15Mn-5Si-9Cr-5Ni-053Nb-0.06C alloy are improved by pre-tensile deformation at room temperature.
2) 90% shape recovery of a 4 % initial strain was obtained when the alloy was pre-extended 12 %, followed by aging at 1070K for 10min.
3) The recovery stress is enhanced with the increase of the amount of pre-extension. By 12% pre-extension, 300 MPa recovery stress was obtained for zero recovered strain and about 170 MPa was attained for 2 % recovered strain. This indicates a possibility for practical application such as pipe joints.

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