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Effects of amount of pre-deformation on precipitation of NbC particles and shape memory effect of Fe17Mn5Si8Cr5Ni0.5NbC alloy during electropulsing treatment

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Abstract: To further enhance shape memory effect (SME) and improve treatment technique in Fe-based shape memory alloys, an Fe17Mn5Si8Cr5Ni0.5NbC alloy was subjected to electropulsing treatment after pre-deformation. The results show that the shape recovery ratio increases with the amount of pre-deformation up to 10%. The maximum shape recovery ratio after electropulsing treatment is 8% greater than that after ageing at 1073 K. The number of NbC particles induced by electropulsing treatment is increased and the size of the particles is decreased with the amount of tensile pre-deformation up to 10%, and NbC carbides are precipitated more quickly through electropulsing treatment than by ageing.

Key words: shape memory alloys; electropulsing treatment; NbC precipitates; ageing; pre-deformation

1 Introduction

Since SATO et al [1] found in 1982 that Fe−Mn−Si alloys exhibited excellent shape memory effect (SME) in single crystal, many studies have been carried out on these alloys because Fe−Mn−Si based shape memory alloys (abbreviated as SMAs) have good workability, good machinability and good weldability. However, Fe−Mn−Si alloys got no important industrial applications. The main reason is that the shape memory effects in Fe−Mn−Si alloys are not as significant as in Ni−Ti alloys, except for some special cases such as single crystals or thin foils [2,3]. To obtain satisfactory shape recovery in Fe−Mn−Si alloys, thermomechanical training, the repetition of deformation and annealing must be conducted [4−6]. Obviously, the cycling prolongs the production time and increases the cost. Furthermore, for complicated shape specimens, the cycling is very difficult to be conducted. Therefore, avoiding cycling treatment and further improving SME of Fe−Mn−Si alloys are crucial for their industrial applications.

KAJIWARA [7], WEN et al [8] and DONG et al [9] found that the additions of Nb/Cr and C elements in Fe−Mn−Si−Cr−Ni based alloys can improve shape recovery ratio because the precipitation of NbC/$Cr_{23}C_6$ carbides can strengthen austenitic matrix and thus suppress the occurrence of permanent deformation. As a result, more strain is afforded by stress-induced martensite transformation, not by plastic slip. Recent work by DONG et al [10] and WANG et al [11] and WEN et al [12−14] further showed that the shape recovery ratio increased with increasing the amount of tensile pre-deformation and reached the maximum value at 10% during ageing after pre-deformation. According to the Orowan theory, for a certain volume fraction of precipitates, the smaller the size of the precipitates is, the greater the strengthening effect is [15]. It has been reported that the electropulsing treatment, as a new potential approach for material processing and preparation, can accelerate the migration of atoms and precipitation of intermetallic compounds [16]. More recent work had also found that the shape recovery ratio of specimens subjected to 5% pre-deformation and 300 V, 1 Hz electropulsing treatment for different time can be further improved and reached the maximum value at 13 s due to the precipitation of smaller NbC carbides in an Fe17Mn5Si8Cr5Ni0.5NbC alloy [17]. Obviously, a

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larger pre-deformation will introduce more defects and thus change the precipitation process of NbC particles during electropulsing treatment, influencing the shape memory effect in FeMnSiCrNiNbC alloys.

It is our aim to study effects of the amount of pre-deformation on precipitation of NbC particles and shape memory effect of an Fe17Mn5Si8Cr5Ni0.5NbC alloy during electropulsing treatment. The results showed that the shape recovery ratio of an Fe17Mn5Si8Cr5Ni0.5NbC alloy increased with increasing the amount of tensile pre-deformation up to 10%, over which it decreased.

2 Experimental

The experimental alloy was prepared by induction melting under an argon atmosphere, using high purity iron, manganese, silicon, chromium, nickel, niobium and graphite. After being homogenized at 1423 K for 15 h, the ingots were hot forged into bars with 15 mm in diameter, and finally cold drawn into wires with 1.6 mm in diameter. The chemical compositions of the alloys are as follows (mass fraction, %): 16.78Mn, 5.50Si, 8.23Cr, 4.77Ni, 0.48Nb, 0.04C and balance Fe. All specimens for measurement were cut from as-cold drawing wires of 1.5 mm in diameter. To eliminate the effect of ε martensite induced by cold drawing and set the specimens in straight shape, the specimens were straightened first and then annealed at 1373 K for 30 min, followed by a water quenching. The quenched specimens were divided into two groups. One group was subjected to electropulsing treatment for different parameters after different amounts of tensile pre-deformation at room temperature on a mechanical testing instrument. The other group was subjected to ageing at 1073 K for different time which is the optimal ageing temperature reported by WANG et al [11] (served as comparison). The electropulsing procedure was performed under ambient conditions, by capacitor discharge (5000 μs, 300 V) with automatic triggering. The current density, J, was 4×10^9 A/m^2, and the pulse duration, t_p, was 400 μs. The shape recovery ratio was determined by bending test as described in Ref. [18]. The bending strain ε is 5%, evaluated by the formula d/D, where d is the diameter of the specimen and D is the diameter of the bending mold.

Observation of microstructure was carried out by JSM−5910LV scanning electron microscope (SEM).

3 Results and discussion

3.1 Effects of electropulsing treatment and amount of tensile pre-deformation on shape recovery ratio

Figure 1 shows the effect of the amount of tensile pre-deformation before electropulsing treatment on shape recovery ratio η of specimens subjected to 300 V, 1 Hz electropulsing treatment for 13 s. η increased sharply with the amount of tensile pre-deformation up to 5%. Over 5%, it increased slowly to the maximum (83%) at 10%, over which the further pre-deformation resulted in degradation of the shape recovery ratio.

Figure 2 shows the effect of operation time t on shape recovery ratio of specimens subjected to electropulsing treatment and ageing after 10% pre-deformation, respectively. For ageing after 10% pre-deformation, η increased gradually from 54.5% to 76.5% with ageing time up to 1800 s. Over 1800 s, the further increase of time had little effect on η. For the electropulsing treatment at 300 V and 1 Hz, η increased sharply with the increase of operation time and at 13 s it reached the maximum value (83%). Over 13 s, η decreased sharply with the increase of operation time.

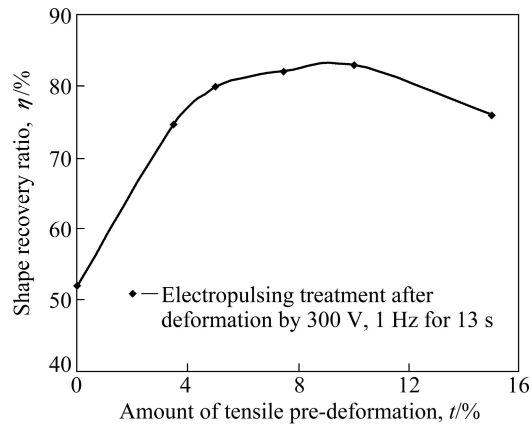

Fig. 1 Effect of amount of tensile pre-deformation on shape recovery ratio

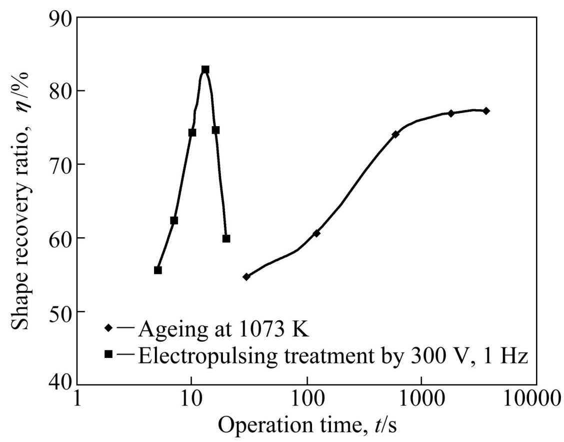

Fig. 2 Effect of operation time on shape recovery ratio of specimens subjected to electropulsing treatment and ageing after 10% pre-deformation

3.2 Effects of electropulsing treatment on precipitation of NbC carbides

Figure 3 shows the SEM micrographs of specimens subjected to 300 V, 1 Hz electropulsing treatment for 13 s

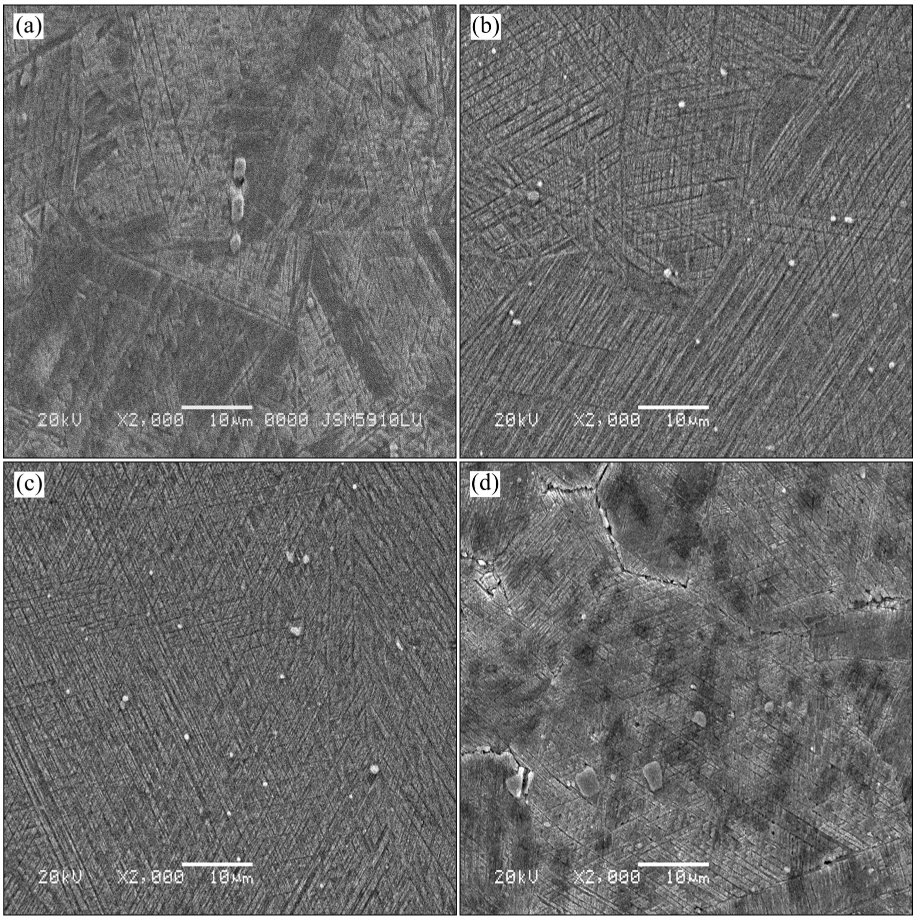

Fig. 3 SEM images of second phases by 300 V, 1 Hz electropulsing treatment for 13 s after different amounts of pre-deformation: (a) 0; (b) 5%; (c) 10%; (d) 15%

after different amounts of tensile pre-deformation. It can be seen obviously that the number of carbides induced by electropulsing treatment was much more and the size of carbides was smaller after 5% pre-deformation than that without pre-deformation. While compared to that after 5% pre-deformation, the number of carbides was increased and the size of carbides was decreased just slightly after 10% pre-deformation. In contrast, the size of some carbides was larger after 15% pre-deformation.

Figure 4 shows the SEM micrographs of specimens subjected to 300 V, 1 Hz electropulsing treatment for different time and ageing at 1073 K for 1800 s after 10% pre-deformation, respectively. It can be seen clearly that carbides induced by electropulsing treatment precipitated more quickly and the number was more and the size was smaller than that by ageing. It was surprising that the size of particles induced by electropulsing treatment for 20 s was much greater than that for 13 s.

Table 1 shows the chemical compositions of second phases by EDS analysis through electropulsing treatment and ageing, respectively. The results showed that second phases were rich in Nb and C, while other elements were much less. Besides, both atomic fraction of Nb and C were close to 50%. Thus, it is doubtless that second phases are NbC.

The precipitation of second phase particles is a process involving atoms diffusion, nucleation and growth. As mentioned in introduction section, electropulsing treatment can accelerate atoms migration. As a result, Nb and C atoms aggregate more easily and quickly than that during ageing. In other words, the number of precipitates as nuclei of NbC in one unit of time during electropulsing treatment is more than that during ageing. However, the growth of the nuclei of NbC is difficult during an extremely short operation time. This is why the smaller NbC particles can be obtained by 300 V electropulsing treatment for 13 s. On the other hand, as a result of accelerating atoms migration by electropulsing treatment, NbC precipitates aggregate, grow up and coarsen more easily and fast. This may be the reason that the size of NbC particles induced by 300 V electropulsing treatment for 20 s was much greater than that for 13 s.

According to the Orowan-Ashby equation, the shear strength increment which causes dislocations to bow out between fine NbC particles can be expressed as [19]:

$$\Delta\tau = \frac{\alpha}{\langle L\rangle}\ln\frac{\langle\gamma\rangle}{b}$$

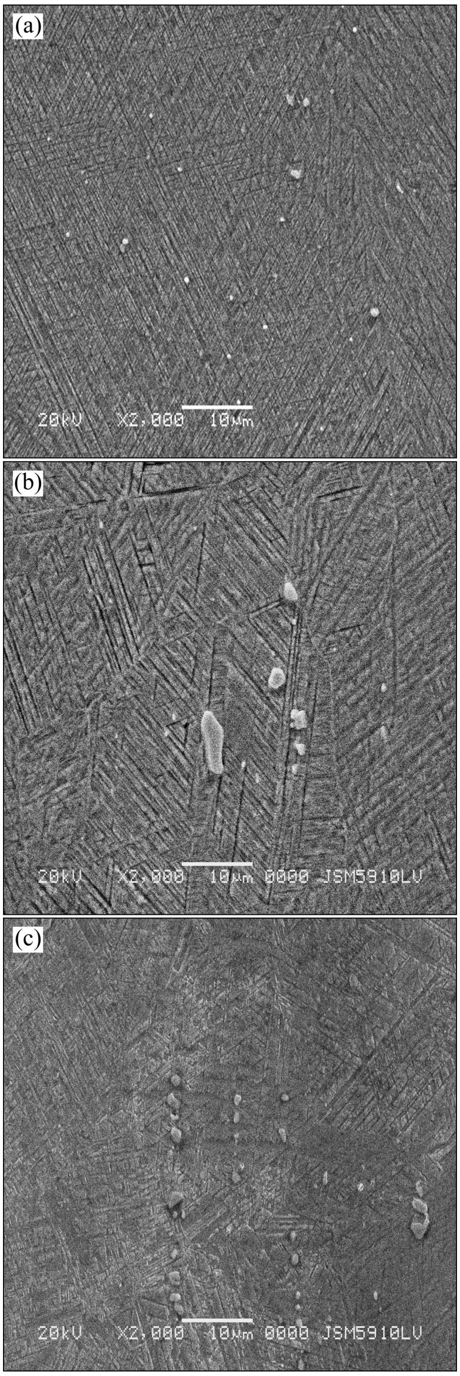

Fig. 4 SEM images of second phases after 10% pre-deformation by 300 V, 1 Hz electropulsing treatment for 13 s (a), by 300 V, 1 Hz electropulsing treatment for 20 s (b) and by ageing at 1073 K for 1800 s (c)

Table 1 Chemical compositions of second phases by EDS analysis through electropulsing treatment and ageing

Processing mode	Mole fraction/%						
	C	Si	Cr	Mn	Fe	Ni	Nb
Electropulsing treatment	46.98	0.13	0.70	1.27	3.46	0.14	47.32
Ageing	47.76	0.33	0.42	0.8	2.3	0.16	48.23

where b is the Burgers vector of the dislocation; $\langle \gamma \rangle$ is the average particle radius; α is a constant. $\langle L \rangle = \frac{4(1-f)\gamma}{3f}$, is the average interparticle spacing, f is the volume fraction of the precipitates. The relationship shows that the greater the volume fraction of the precipitates and/or the smaller the radius of the precipitates, the greater the strengthening effect. This is why the better SME can be obtained by 300 V electropulsing treatment for 13 s after 10% pre-deformation. Whereas, as a result of coarsening of NbC particles, the shape recovery ratio of specimens subjected to 300 V electropulsing treatment decreased sharply over 13 s.

When pre-deformation is carried out at room temperature, lots of γ/ε interfaces are produced where NbC particles will nucleate preferentially due to the remarkable decrease of activation energy for precipitate nucleation [14]. With the amount of tensile pre-deformation up to 10%, the number of γ/ε interfaces can be increased markedly. As a result of the increase of nucleation site, the number of NbC particles is more and their size is smaller. When the amount of pre-deformation is larger, the crosses and collisions between different direction ε martensite bands will occur seriously and at the crossing areas, the plastic deformation of the martensite bands will be induced. The plastic deformation increases the energy of the crossing areas and thus makes NbC particles preferentially nucleate and grow up more easily. As a result, the size of NbC particles is larger after 15% pre-deformation. This explains why the shape recovery ratio decreases when the amount of tensile pre-deformation is beyond 10%.

4 Conclusions

With the amount of tensile pre-deformation up to 10%, the number of NbC particles is increased and the size of the NbC particles is decreased gradually during electropulsing treatment in an Fe17Mn5Si8Cr5Ni0.5NbC alloy. Besides, compared to ageing, after 10% pre-deformation, electropulsing treatment can make NbC carbides precipitate more quickly and the number of NbC particles is more and the size of the particles is smaller in the pre-deformed alloy. Therefore, the better SME can be obtained.

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电脉冲处理对不同预变形量 Fe17Mn5Si8Cr5Ni0.5NbC 合金 NbC 析出相与形状记忆效应的影响

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摘 要：为进一步提高铁基形状记忆合金的记忆效应并改善处理技术，研究了电脉冲处理对不同预变形量 Fe17Mn5Si8Cr5Ni0.5NbC 合金性能的影响。结果表明：当预变形量低于 10%时，电脉冲处理后合金的形状回复率随着预变形量的增大而增大，且最大值比 800 °C 热处理的合金高 8%左右。与热处理状态相比，当预变形量低于 10%时，随着预变形量的增大，电脉冲处理诱导出 NbC 颗粒的数量更多、尺寸更小、析出速度更快，进而有效改善合金的记忆性能。

关键词：形状记忆合金；电脉冲处理；NbC 析出；时效；预变形

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