Study on martensitic transformation of Fe-based alloys with low relaxation based on constant strain constraint

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Abstract—The paper analyzed the martensite transformation characteristic of Fe-Mn-Si alloy by XRD analysis and tensile test under different deformation condition. In this study, the amount of ε martensite of Fe-17Mn-5Si-10Cr-5Ni alloy was lower than that of Fe-17Mn-5Si-2Cr-2Ni-1V alloy under the same deformation condition, but the change rate of ε martensite was much higher than that of Fe-17Mn-5Si-2Cr-2Ni-1V alloy, and the stress relaxation rate of the both alloys increased with the increase of the stop loading time, and the relaxation rate of the Fe-17Mn-5Si-10Cr-5Ni alloy was obviously higher than that of Fe-17Mn-5Si-2Cr-2Ni-1V alloy. (Abstract)

1. INTRODUCTION
Fe based memory alloys, especially Fe-Mn-Si based memory alloys, have attracted much attention due to their low cost, good mechanical properties and good cutting performance. However, the shape memory effect of Fe-Mn-Si based memory alloys is poor, and the recovery rate is only 2%-3%, and the restoring force is easy to relax at room temperature. Therefore, how to improve the shape memory effect and stress relaxation characteristics has become the focus of research at home and abroad. In this paper, according to the “equilibrium carbon” principle of high speed steel[1], element V is added to Fe-Mn-Si based alloys, and the C content in the alloy is appropriately increased, and a low-relaxation shape memory alloy is signed to solve the problem of low-temperature stress relaxation of Fe-Mn-Si based alloys. The stress relaxation characteristic and martensite transformation of Fe-Mn-Si based alloys with low relaxation under different deformation conditions were analyzed by XRD analysis and tensile test.

2. EXPERIMENTAL PROCESS
Fe-16.9Mn-4.49Si-10.29Cr-5.30Ni-0.07C (wt%) (A for short) and Fe-17.5Mn-4.49Si-3.21Cr-2.95Ni-0.27C-1.44V (wt%) (B for short) were prepared. The alloy samples were smelted in the vacuum medium frequency melting furnace and cast ingots were moulded. After being homogenized at 1200 °C and heated to 1100 °C for 1 hour, the alloy samples were hot-forged into a 15 mm×15 mm blank. The A and B alloy samples were processed by wire cutting, and the effective size was 28 mm×4 mm×1 mm. The sample was treated with argon gas in a tubular resistance furnace for 1 hour heat preservation at 1000 °C. After solid solution treatment, B alloy was annealed at 500°C for 10min. The tensile
deformation at room temperature was completed on the MTS880 material test machine with a strain rate of \(1.19 \times 10^{-3} \text{s}^{-1}\). The specimens were stretched according to three deformation processes:

1. After stretching to 5%, 7% and 9% of the pre-deformation, unload directly.
2. After stretching to 5%, 7% and 9% of the pre-deformation, keep the chuck still, stop loading for 10 min and unload.
3. After stretching to 5%, 7% and 9% of the pre-deformation, keep the clamping head still, stop loading for 60 min and unload.

X-ray diffraction measurement was carried out on the Japanese physics motor D/max-3b X-ray diffractometer, the test condition was CuKα ray radiation, the working voltage was 35 kV, the current was 100 mA, and the scanning speed was 2 °/ min. The specimens are directly drawn. In order to remove the surface dirt, it was necessary to clean with royal water.

Pre-strain εp and shape recovery rate η respectively (εp = \(\frac{(L1 - L0)}{L0}\) × 100%, η = \(\frac{(L1 - L2)}{(L1 - L0)}\) × 100%, among them \(L0, L1\) for each pre-strain before and after the length of the sample, \(L2\) for the deformation and heated to 500 °C the length of the insulation after 5 min and then cooling to room temperature.

3. RESULT AND DISCUSSION

Figure 1 showed the XRD of B alloy samples with a pre-deformation of 5%, which were directly unloaded, stopped loading for 10 min and stopped loading for 60 min. With the increase of stopping time, the peak of (10.1) increased significantly, indicating that the stress-induced martensitic \(\gamma \rightarrow \epsilon\) transformation occurred in the alloy, which was beneficial to the shape memory effect. (110) \(\alpha'\) peak exists in the alloy during directly unloading, and the (110) \(\alpha'\) peak increases with the increase of stop loading time. There were two views on the origin of the \(\alpha'\) martensite: one was the \(\alpha'\) martensite formed by the intersection of \(\epsilon\) martensite plates when the \(\epsilon\) martensite transition was induced under large stress [2]; another thought was that stress directly induces \(\gamma \rightarrow \epsilon\) martensitic transformation [3]. The presence of \(\alpha'\) martensite introduced irreversible plastic deformation which did not produce shape memory effect.

A direct comparison method [4] was used to quantitatively calculate the \(\gamma, \epsilon, \alpha'\) phase of alloy A and B samples with a pre-deformation amount of 5% under direct unloading, stopped loading for 10 min and stopped loading for 60 min, as shown in table 1.

With the increase of stopped loading time, the volume of \(\epsilon, \alpha'\) martensite both increased, while the volume of \(\gamma\) austenite decreased in alloys A and B, which was consistent with XRD result. At the same state, the \(\epsilon\) martensite volume in B alloy was more than that of A alloy, and the change rate of \(\epsilon\) martensite volume in B alloy was much lower than that of A alloy. This was because the addition of V and C elements in B alloy strengthens the parent phase, hindered the growth and intersection of \(\epsilon\) martensite, and made it difficult to transform the stress-induced \(\gamma \rightarrow \epsilon\) martensitic.
For B alloy, the increase rate of the stress-induced ε martensitic volume was 0.43% at stopped loading time of 0 to 10 min, and the increase rate of the stress induced ε martensitic was 0.35% at stopped loading time of 10 to 60 min. However, during the stopping process, the volume of α’ martensitic was continuous increases. It can be concluded that, the increase of the amount of ε martensitic was inhibited by the increase of the amount of α’ martensitic under the constant strain suspending loading, which maked the increase rate of the ε martensitic slow.

Table 1 Volume fraction of γ, ε and α’ phases of alloy A and B samples with pre-deformation 5% under direct unloading, stopped loading for 10 min and stopped loading for 60 min

| NO. Deformation condition   | γ%  | ε%  | α’% |
|-----------------------------|-----|-----|-----|
| Directly unloading          | 18.63 | 79.02 | 2.35 |
| Stopped loading for 10 min  | 15.57 | 82.04 | 2.39 |
| Stopped loading for 60 min  | 15.02 | 82.56 | 2.42 |
| Direct unloading            | 6.09  | 87.93 | 5.98 |
| Stopped loading for 10 min  | 4.82  | 88.31 | 6.87 |
| Stopped loading for 60 min  | 4.21  | 88.62 | 7.17 |

For B alloy, the increase rate of the stress-induced ε martensitic volume was 0.43% at stopped loading time of 0 to 10 min, and the increase rate of the stress induced ε martensitic was 0.35% at stopped loading time of 10 to 60 min. However, during the stopping process, the volume of α’ martensitic was continuous increases. It can be concluded that, the increase of the amount of ε martensitic was inhibited by the increase of the amount of α’ martensitic under the constant strain suspending loading, which maked the increase rate of the ε martensitic slow.

Figure 2 showed the effect of stopped loading time on the stress relaxation rate of alloy A samples with different pre-deformation. With the increase of stopped loading time, the stress relaxation rate of A alloy increased significantly. The stress decreased sharply with first 5 min, and the rate was faster. In other words, the martensite transformation was completed within the first 5 min, which was also confirmed with the characteristics of martensite transformation phase transition that was instant nucleation, rapid growth, and limited size [5]. Figure 3 showed effect of stopped loading time on stress relaxation rate in B alloy samples with different pre-deformation.

Figure 2. Effect of suspending loading time on stress relaxation rate in A alloy specimens with different pre-strains

Figure 3. Effect of stopped loading time on stress relaxation rate in B alloy specimens with different pre-strains
The change rule of the stress relaxation rate of B alloy was similar to that of A alloy. Compared with that of A alloy, it was found that at the first 5 min, the stress relaxation rate of alloy B with the pre-deformation amount of 5% was 2.63% lower than that of alloy A, because B alloy had more carbon than A alloy. The addition of V and C elements in B alloy strengthened the parent phase, which could increase the resistance of the incomplete dislocation in the parent phase along the crystal surface, making it difficult to the transformation of the matrix, thus effectively inhibiting the stress-induced martensitic transformation and reducing the stress relaxation rate. The stress relaxation rate of B alloy with the pre-deformation of 9% was lower than that of 5% and 7%. This was because the irreversible dislocation slip deformation in the parent phase occupied a greater advantage at a higher deformation amount [6]. Therefore, at a higher stress level, the deformation of the alloy was mainly caused by dislocation proliferation and movement, and the dislocation density increased rapidly with the increase of deformation, thus forming "dislocation entanglement" [7].

4. CONCLUSION
1. Under the same deformation condition, the ε volume of B alloy was more than that of A alloy, and the change rate of ε volume was much lower than that of A alloy. Because the addition of V and C elements in B alloy strengthened the parent phase could increase the resistance of the incomplete dislocation in the parent phase along the crystal surface.

2. With the increase of stopped loading time, the stress relaxation rate of A and B alloys increased significantly, and the relaxation rate of B alloy was significantly lower than that of A alloy under the same conditions. This is because the addition of V and C elements to B alloy strengthened the parent phase, effectively inhibited the stress-induced martensitic transformation, and reduced the stress relaxation rate.

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