Study on the reverse martensitic transformation of Fe-Mn-Si alloy based on constraint state

Linlin Liu*, Yijian Zhao, Tianxiang Zhao
School of Mechanical Engineering; DaLian JiaoTong University, Da Lian Liao Ning 116028, China;
Corresponding Author: LIU Lin-lin; email: Liulinlin@djtu.edu.cn;

Abstract. The constrained stress-induced ε reverse martensitic transformation characteristic of Fe-Mn-Si alloy was studied by X-Ray Diffraction (XRD) and metallographic analysis. The results show that the origination temperature (A_s) of the constrained reverse transformation of Fe-Mn-Si alloy in cement matrix, compared with the unconstrained alloy, was immovability, the pause temperature (A_f) was elevated, and the ε reverse martensitic transformation became slower under the same recovery temperature. At the same time, the amount of the constrained stress-induced ε reverse martensitic transformation decreased and the reverse transformation temperature zone broadened with the increasing degree of prestrain.

1. Introduction
Shape memory alloy (SMA) is one of the first driving elements used in civil engineering. It can be used in various deformation forms to recover large deformation. SMA can produce a large driving force under limited recovery. It has good fatigue resistance and is easy to be combined with structural materials such as concrete and steel. The elastic modulus changes with the change of phase transformation state[1-4]. At present, the expensive NiTi alloy is used in most of the shape memory alloys used in composite structures, but its application is limited. The driving effect of Fe-Mn-Si SMA on the structure of composites is based on the reverse transformation of ε martensitic in the constrained state, which is rarely studied. In this paper, X-ray diffraction (XRD) and metallographic observation have been used to study the reverse transformation of stress induced martensitic in pre-strained Fe-17Mn-5Si-10Cr-5Ni SMA.

2. Test materials and methods
The test material was Fe-17Mn-5Si-10Cr-5Ni(wt%) alloy (A alloy for short). After the ingot was demolded, it was first homogenized at 1200℃, then heated to 1100℃ and held for 1h, and then hot forged into 15mm×15mm blank. The alloy samples of 2mm×2mm×260mm size were processed by wire cutting, and the samples were treated with solid solution at 1000℃×1h under the protection of argon in a tubular resistance furnace. The sample preparation process under the constraint of cement matrix is as follows: cement is portland 525 # cement. The samples were stretched to 3.36%, 4.73% and 7.94% at room temperature (293K), and one of them was put into a fixed size mold. The mold was filled with evenly mixed cement slurry (water-cement ratio was 0.45), and the cement surface was kept wet after standing for 24 hours. After demould, a cement beam with a size of 40mm×40mm×280mm was obtained. After curing the cement beam at room temperature for 7 days and drying for 3 days, the cement beam and the alloy specimen were evenly cut into 30mm short pieces and heated to 50℃, 100℃, 200℃, 250℃, 300℃, 350℃, 400℃, 500℃ for 1h. After cooling at...
room temperature, the test alloy sample in the cement beam section was taken out to obtain the test alloy recovery sample under the constraint of cement matrix.

The X-ray diffraction measurement was carried out on the D/ MAX-3B X-ray diffractometer of Nippon Electric Motor. The test conditions were CuKα radiation, the working voltage was 35kV, the current was 100mA, and the scanning speed was 2°/min.

The metallographic observation was Olympus G×51 microscope. In order to obtain better corrosion effect, the ratio of 3mlHf +50mlH₂O₂+10mlH₂O solution was used for chemical polishing to remove the stress-induced martensite layer caused by grinding on the surface, and then 3gCuSO₄+10mlHCl+30mlH₂O solution was added for corrosion.

3. Experimental results and discussion

Fig.1 shows the X-ray diffraction spectrum of the unconstrained state of A alloy with pre-deformation of 3.36% at different heating temperatures. It can be seen from the figure that the peak of (200)γ becomes stronger and the peak of (10.1)ε decreases with the increase of temperature. After heating at 250℃, the ε martensitic in the microstructure disappears completely, and the alloy becomes a single γ austenite, which indicates that the γ → ε martensitic reverse transformation occurs in the alloy specimen during heating. At the same time, macroscopic shape recovery was observed. It is clearly revealed that the shape memory properties of Fe-Mn-Si alloy depend on the γ → ε martensitic transformation and its reverse transformation.

As can be seen from the figure, when the temperature rises to 250℃, peak (10.1) ε still exists and continues to decrease with the increase of temperature. When heated to 500℃, martensitic still exists, that is to say, when the temperature rises to 500℃, the reverse transformation still continues, indicating that the end temperature of the reverse transformation of the alloy specimen under the constraint of cement matrix is higher than 500℃. Compared with Fig. 1 and Fig. 2, it is found that the end temperature of the reverse transformation of the cement matrix constrained alloy sample with a pre-deformation of 3.36% is higher than that of the unconstrained state. That is to say, when the end temperature of the reverse transformation of the unconstrained state alloy is above Aᵣ, the reverse transformation of the constrained state is still going on and the temperature range of the constrained state transformation is expanded. The experimental results show that with the increase of pre-deformation, the end temperature of reverse transition increases, and the reverse transformation rate slows down. This is because when the pre-deformation of the alloy increases, the martensitic with different orientation will cross collision, and the interface of the cross collision will be distorted, which is easy to generate stress concentration, leading to the slip and increment of the whole
dislocation [5]. The increase of total dislocation will greatly hinder the reverse motion of Shockley incomplete dislocation, making martensitic stable and requiring higher temperature to reverse change. At the same time, the reversibility of martensitic reversal at the intersection is reduced. Although the amount of stress induced martensitic is large during large deformation, the stability of martensitic and the decrease of reversibility make the reversal rate of martensitic heating in the alloy samples with large deformation lower than that with small deformation.

Fig. 3 shows the X-ray diffraction spectrum of the unconstrained state of A alloy samples with different deformations at 350°C. As can be seen from the figure, when heated to 350°C, martensitic no longer exists in 3.36% and 4.73% of the pre-deformed alloy samples, indicating that the reverse transformation of the alloy has completely ended at this time, while the peak (10.1) ε still exists in 7.94% of the predeformed alloy samples. It can be concluded that with the increase of predeformation, the martensitic volume increases and the reverse transformation end temperature Af increases. At the same time, it can also be seen from the figure that, with the increase of predeformation, the austenite content of the phase gradually decreases, while the martensitic volume slightly increases.

![Fig. 3 X-ray diffraction patterns of unconstrained alloy samples with different deformations at 350°C](image1)

![Fig. 4 X-ray diffraction patterns of constrained alloy samples with different deformations at 400°C](image2)

Fig. 3 shows the X-ray diffraction spectrum of the constrained A alloy samples with different deformations at 400°C. Can be seen from the figure, the amount of parent phase austenite gradually reduce with increasing the predeformation in the constraint cement matrix, namely, the amount of the martensitic reverse variables is reduced, and the reverse phase transition end temperature Af rise with the increase of predeformation should be variable, shows that the reverse phase transformation temperature range (Af - As) with the increase of amount predeformation and broadening.

Fig. 5 shows the microstructure of the constrained alloy sample with predeformation 4.73% after recovery annealing at 250°C and 400°C, respectively. The white matrix is γ austenite, and martensite is distributed in different quantities on the matrix.

Region A is acicular martensitic bundles with straight boundary in Fig. 5 (a), fine size and parallel arrangement, indicating that the martensite formed in a grain is a variant of the same phase on the {111} habitus plane. These martensitic bundles run through the whole grain and terminate at grain boundary. The shape memory effect of the alloy is essentially caused by the ε→γ martensitic reverse transformation during heating. The chance of cross crossing between martensities are greater when the deformation is larger and the stress induced martensitic is more[6].As shown in Region B in Fig. 5 (a), stress concentration is generated at the intersection, which increases the chance of slip and appreciation of the whole dislocation. The existence of the whole dislocation will greatly hinder the reverse movement of the incomplete dislocation and stabilize the martensitic, and a higher temperature is required to reverse change. This is the reason why the end temperature of ε→γ inverse martensite transformation Af increases.
As can be seen from Fig. 5 (a), after the recovery annealing at 250°C, the number of residual lamellar martensitic is still relatively large, indicating that the reverse transformation degree of $\varepsilon \rightarrow \gamma$ martensitic is low. With the increase of recovery temperature, the reverse martensitic transformation continues, and the number of martensitic further decreases. Fig. 5 (b) shows the metallographic structure of the alloy sample in the constrained state after recovery annealing at 400°C. Compared with Fig. 5 (a), the amount of martensite has been significantly reduced and the microstructure has been more uniform, so the shape memory effect tends to be stable.

![Microstructure of constrained Fe-Mn-Si alloy specimens with 4.73% at the recovery temperature of 250 °C and 400 °C](image)

(a) at 250 °C recovery temperature; (b) at 400 °C recovery temperature

When the stress (strain) on the Fe - Mn - Si shape memory alloy, only experience preferred orientation of martensitic change along the direction Schmid factor biggest annexation adverse orientation variations of priority grew up, absorb local energy as a mechanical driving force to promote martensitic phase transformation, slows down the adjacent grains because elastic-plastic mismatch caused by stress concentration [7]. Variations between mutual annexation and variation of the quantity and the decrease of martensitic lath interface, effectively release the elastic strain energy stored in the martensitic variants, and the elastic strain energy is a driving force of the inverse martensitic phase transformation, the inverse phase transformation driving force is reduced, further increases the friction energy dissipation, lead to martensitic stabilization occur, so that the constraint state inverse martensitic phase transformation was postponed to a temperature higher than the original constraint state temperature, end temperature rise, which broadens the constraint state inverse phase transition temperature range. However, for the unconstrained reverse phase transformation of the alloy, the grain only forces along the grain boundary. During the heating process, the driving force required by the martensitic transformation is unchanged, and the end temperature of the reverse phase transformation does not change. Therefore, the end temperature of the reverse phase transformation is low, and the corresponding temperature range of the reverse phase transformation is narrow.

4. Conclusion

Compared with the unconstrained alloy, the initial temperature of the reverse phase transformation of the alloy under the constraint of cement matrix is unchanged, while the end temperature $A_f$ is increased. The reverse phase transformation rate is slower, and the larger the pre-strain is, the higher the $A_f$s is. The reversal variables of stress-induced martensitic under constraint state are reduced, and the temperature range of reverse phase transition ($A_f-A_s$) is widened.

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