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Abstract

The shape memory effect and microstructure of solution-treated Fe-(14~21)Mn-5.5Si-8.5Cr-5Ni alloys with three different Mn contents were investigated before and after deformation at 10 K above the martensitic transformation start temperature (Ms+10 K) using OM, EBSD, XRD, TEM, and SQUID. The results indicate that the shape memory effect of the solution-treated Fe-(14~21)Mn-5.5Si-8.5Cr-5Ni alloys increases with increasing Mn content. This is attributed to: on the one hand, the difference between the austenite yield strength and the critical stress for stress-induced ϵ martensite increases with increasing Mn content, which enhances the resistance of austenite to plastic deformation; on the other hand, the increased Mn content reduces the width of stress-induced ϵ martensite and suppresses the formation of α' martensite, thereby improving the reversibility of stress-induced ϵ martensite.

Full Text

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Influence Mechanism of Mn Content on the Shape Memory Effect in Fe-Mn-Si-Cr-Ni Alloys*

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Abstract OM, EBSD, XRD, TEM and SQUID were used to investigate the shape memory effect and microstructures of three solution-treated Fe-(14-21)Mn-5.5Si-8.5Cr-5Ni alloys with different Mn contents before and after deformation at 10 K above the martensitic transformation start temperature M_s (M_s+10 K). The results show that the shape memory effect of solution-treated Fe-(14-21)Mn-5.5Si-8.5Cr-5Ni alloys increases with increasing Mn content. This is because, on the one hand, the difference between the yield strength of austenite and the critical stress for stress-induced ε-martensite increases with increasing Mn content; that is, increasing the Mn content enhances the ability of austenite to resist plastic deformation. On the other hand, increasing the Mn content reduces the width of stress-induced ε-martensite and suppresses the introduction of α'-martensite, thereby improving the reversibility of stress-induced ε-martensite.

Key words shape memory alloy, Mn, stress-induced ε-martensite, slip

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EFFECT MECHANISM OF Mn CONTENTS ON SHAPE MEMORY OF Fe-Mn-Si-Cr-Ni ALLOYS

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ABSTRACT Fe-Mn-Si base shape memory alloys (SMAs), as compared with Ni-Ti and Cu base SMAs, have attracted much attention since the 1980s due to their promising advantages, such as low cost, good workability and weldability. However, the recovery strain of polycrystalline Fe-Mn-Si base SMAs is only about 2%–3% except single crystals and ribbons ones. At the present time, in order to enhance the recovery strain of this kind of alloys, some methods such as thermo-mechanical training, ausforming and thermo-mechanical treatment are used. In recent years, the research group had prepared training-free cast Fe-Mn-Si base alloys showing an excellent shape memory effect (SME). Unfortunately, the grains of cast Fe-Mn-Si base alloys are coarse, certainly leading to low yield strength and recovery stress. Many factors affecting the shape memory effect, such as alloy elements, the amount of pre-strain, deformation temperatures, annealing treatments and the training, have been studied. However, there is a debate on the effect of Mn contents on the shape memory effect of Fe-Mn-Si base alloys. The aim of this work is to clarify the debate, shape memory effect and microstructures of solution treated Fe-(14–21)Mn-5.5Si-8.5Cr-5Ni alloys were investigated by OM, EBSD, XRD, TEM and SQUID before and after deformation at 10 K higher than their start temperature of martensitic transformation (M_s+10 K). The result showed that the shape memory effect of solution treated Fe-(14–21)Mn-5.5Si-8.5Cr-5Ni alloys increased with the Mn contents. There are two reasons for this result. One is that the difference value between austenitic yield strength and critical stress of stress-induced ε martensite increased with the Mn contents. In other word, the ability resisting plastic deformation was

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improved by increasing the Mn contents. The other is that the reversibility of ε martensite reverse transformation was enhanced by increasing the Mn contents because the width of stress-induced ε martensite decreased while the α' martensite was difficult to be introduced with increasing the Mn contents.

KEY WORDS shape memory alloy, Mn, stress-induced ε martensite, slip

Fe–Mn–Si-based shape memory alloys have attracted considerable attention in recent years because of their low cost, ease of processing, and excellent mechanical properties[1–12]. Studies have shown that single-crystal and thin-strip Fe–Mn–Si-based alloys exhibit excellent shape memory effects[1,2], whereas untreated polycrystalline Fe–Mn–Si-based alloys show poor shape memory effects, with recoverable strain below 3%. To improve the shape memory effect of Fe–Mn–Si-based alloys, extensive studies have been conducted on the effects of alloying[7,8], thermomechanical cyclic training[9–10,13], high-temperature deformation heat treatment of austenite[14], and precipitation of a second phase[5,15] on their shape memory effect. The results show that thermomechanical cyclic training is currently the most effective method for improving the shape memory effect of Fe–Mn–Si-based alloys, increasing the recoverable strain of the alloy to 4%–5%. However, this treatment process is complicated and increases manufacturing costs. In recent years, our group has prepared training-free cast Fe–Mn–Si-based shape memory alloys by casting followed by annealing[16], for which the recoverable strain exceeds 6%. Nevertheless, cast alloys suffer from low yield strength and low recovery stress. Therefore, how to prepare training-free Fe–Mn–Si-based alloys with both excellent shape memory effect and good mechanical properties remains an urgent problem to be solved.

Mn is one of the basic constituent elements of Fe–Mn–Si-based alloys. However, the mechanism by which Mn affects their shape memory effect remains unclear. Otsuka et al.[17] investigated the shape memory effect of Fe–(14–22)Mn–5Si–8Cr–5Ni (mass fraction, %, the same below) alloys during deformation at room temperature. Their results showed that the more stress-induced ε martensite formed, the better the shape memory effect of the alloy. Inagaki and Inoue[18] also studied the shape memory effect of Fe–(10–28)Mn–6Si–9Cr–6Ni alloys during deformation at room temperature. Their results likewise showed that, when the Mn content was between 18% and 28%, the more stress-induced ε martensite formed, the better the shape memory effect of the alloy. However, they also found that, when the Mn content was between 14% and 18%, the more stress-induced ε martensite formed, the poorer the shape memory effect of the alloy.

Inagaki and Inoue[18] believed that this result was due to the presence of thermally induced ε martensite in the alloy when the Mn content was below 18%, which was unfavorable for the reverse transformation of stress-induced ε martensite. However, Federzoni and Guénin[19] found that, when the amount of ther-

mally induced ε martensite was below 10%, it was beneficial to improving the shape memory effect of Fe-Mn-Si-based alloys. In addition, Mn can alter the start temperature M_s for the transformation from austenite γ to ε martensite in Fe-Mn-Si-based alloys[20]. To avoid the influence of thermally induced ε martensite, the present work investigated the shape memory effect and microstructure of Fe-(14-21)Mn-5.5Si-8.5Cr-5Ni alloys with different Mn contents after deformation at 10 K above M_s (M_s + 10 K), thereby clarifying the mechanism by which Mn content affects the shape memory effect of Fe-Mn-Si-Cr-Ni alloys.

1 Experimental Method

The experimental alloys were prepared using industrial-purity Fe, electrolytic Mn, metallic Si, metallic Cr, and electrolytic Ni as raw materials. They were melted in a medium-frequency induction furnace under Ar protection in vacuum and then cast into ingots. The ingots were homogenization-annealed at 1373 K for 12 h and then hot-forged into slabs, which were subsequently hot-rolled into plates. Finally, the plates were cold-rolled with 20% deformation into plates 2.2 mm thick. The plates were then wire-cut into the specimens required for the experiments. The wire-cut specimens were finally solution-treated at 1373 K for 30 min and water-quenched. The chemical compositions of the alloys are shown in Table 1.

The transformation temperatures of the solution-treated alloys were determined from resistivity-temperature curves, and the results are shown in Table 1. According to the measured transformation temperatures, bending was used to characterize the shape memory effect of the alloys after deformation at 10 K above the ε martensite transformation start temperature M_s (M_s + 10 K)[21]. The bent specimens were heated at 873 K for 5 min for recovery. It should be noted that the M_s of the 14Mn alloy is higher than room temperature. To avoid the introduction of thermally induced ε martensite, after solution treatment the specimen was directly quenched into hot water at 337 K to test its shape memory effect. Tensile deformation of dog-bone specimens was carried out in high- and low-temperature environmental chambers using an RGM-4300 universal testing machine,

Table 1 Chemical compositions of Fe-Mn-Si-Cr-Ni alloys and phase transformation temperatures of solution-treated alloys

Alloy	Mn	Si	Cr	Ni	C	Fe	M_s	A_s	A_f
	Mass fraction / %						Transformation temperature / K		
14Mn	14.81	5.63	8.72	5.48	0.006	Bal.	327	345	405
18Mn	18.81	5.61	9.31	5.36	0.010	Bal.	235	336	385
21Mn	21.63	5.60	9.32	5.38	0.015	Bal.	220	328	357

Note: M_s—start temperature of martensite transformation; A_s—start temperature of austenite transformation; A_f—finish temperature of austenite transformation.

The nominal yield strength $\sigma_{0.2}$ of the alloys between 230 and 473 K was characterized. The tensile rate was 0.5 mm/min.

Metallographic observation was carried out on an OLYMPUS GX51 optical microscope (OM). The different phases in the alloys were characterized by the color OM image corrosion method. The etchant for color OM images was an aqueous solution of 1.2% $K_2S_2O_5$ + 0.5% NH_4HF_2 (mass fraction). In the color OM images, γ austenite appears in standard color, while ε martensite appears as black striations and white plates. The grain size of the alloys was characterized using a JSM 6500F field-emission scanning electron microscope (SEM) equipped with a TSL-OIM electron backscatter diffraction system (EBSD). The width of ε martensite in the color OM images was statistically measured using Image analysis FIVE metallographic analysis software; at least 500 martensite plates were counted. An X' Pert Pro MPD X-ray diffractometer (XRD) was used to characterize the diffraction peaks of ε martensite $(10\bar{1}0)$ and $(10\bar{1}1)$, as well as austenite (111), (200) and (220). The volume fraction of stress-induced ε martensite was quantitatively calculated based on the multi-peak method. The target was Cu, and the scanning rate was 2 °/min. The microstructure of the alloys was observed on a Tecnai F20 transmission electron microscope (TEM). TEM specimens were mechanically thinned and then prepared by electrolytic twin-jet perforation. The electrolyte was H_2SO_4:CH_3OH = 1:4 (volume fraction). An MPMS-7T superconducting quantum interference device (SQUID) was used to measure the magnetic saturation intensity of the alloys after different amounts of deformation under 188.4 A/m, thereby characterizing the evolution of α' martensite. In Fe-Mn-Si-based shape memory alloys, α' martensite is a magnetic phase; therefore, changes in the magnetic saturation intensity of the alloys can reflect changes in the amount of α' martensite.

2 Experimental Results

2.1 Effect of Mn Content on the Memory Effect

Figure 1 shows the recovery strain of three alloys with different Mn contents after different amounts of deformation at M_s+10 K. When the deformation was less than 6%, the recovery strain of all three alloys increased with increasing deformation. After the deformation exceeded 6%, the recovery strain of the 14Mn alloy increased slightly, reached a maximum near 8% deformation, and then decreased; the recovery strain of the 18Mn alloy remained unchanged; whereas the recovery strain of the 21Mn alloy continued to increase, reaching 3.7% near 12% deformation. It is worth noting that, under the same deformation, the recovery strain of the alloys increased with increasing Mn content.

2.2 Effect of Mn Content on Stress-Induced ε Martensitic Transformation

Figure 2 shows color OM images of three alloys with different Mn contents. The M_s of the 14Mn alloy (327 K) is higher than room temperature (293 K), so thermally induced ε martensite (white) is generated in the room-temperature microstructure. The M_s values of the 18Mn and 21Mn alloys are both lower than room temperature (Table 1), so their room-temperature microstructures

are single-phase austenite. EBSD was used to characterize the grain sizes of the solution-treated 14Mn, 18Mn and 21Mn alloys, which were 100.7, 97.6 and 104.4 μm, respectively. Thus, the grain sizes of the three alloys are very close.

Figure 3 shows color OM images of three alloys with different Mn contents after different amounts of deformation at $M_s + 10$ K. It can be seen that, in the three alloys with different Mn contents, the amount of stress-induced ε martensite increases as the deformation increases. For the 14Mn and 18Mn alloys, black granular phases appear at intersections of ε martensite, whereas in the 21Mn alloy this black granular phase is difficult to observe. The literature [12,22] has clearly shown that this black granular phase is α' martensite.

Figure 4 shows the XRD spectra and the volume fraction of stress-induced ε martensite for solution-treated 14Mn, 18Mn and 21Mn alloys after different amounts of deformation at $M_s + 10$ K. The results show that, at relatively large deformations of 7% and 9%, distinct α' martensite peaks appear in the 14Mn and 18Mn alloys.

(Figure: Fig.1 Effect of deformation strains on the recovery strain of solution treated 14Mn, 18Mn and 21Mn alloys deformed at their $M_s + 10$ K)

Fig. 1 Effect of deformation strains on the recovery strain of solution treated 14Mn, 18Mn and 21Mn alloys deformed at their $M_s + 10$ K

(Figure: Fig.2 Color OM images of solution treated 14Mn (a), 18Mn (b) and 21Mn (c) alloys)

Fig. 2 Color OM images of solution treated 14Mn (a), 18Mn (b) and 21Mn (c) alloys

(Figure: Fig. 3)

Fig. 3 Color OM images of solution treated 14Mn (a, d, g), 18Mn (b, e, h) and 21Mn (c, f, i) alloys subjected to deformation strains of 4% (a–c), 7% (d–f) and 9% (g–i) at their $M_s + 10$ K

(Fig. 4a and b); however, no α' martensite peaks appear in the 21Mn alloy (Fig. 4c). As shown in Fig. 4d, after the same amount of deformation at $M_s + 10$ K, the volume fraction of stress-induced ε martensite in the alloy decreases with increasing Mn content.

To accurately characterize the amount of α' martensite introduced during deformation, SQUID was used to characterize the saturation magnetization of the three alloys with different Mn contents after different amounts of deformation at $M_s + 10$ K, as shown in Fig. 5. When the deformation strain is 4%, only the saturation magnetization of the 14Mn alloy increases, while those of the 18Mn and 21Mn alloys remain essentially unchanged. After the deformation strain exceeds 4%, the saturation magnetization of the 14Mn alloy continues to increase, that of the 18Mn alloy increases slowly, and that of the 21Mn alloy still remains unchanged. The above SQUID results indicate that the amount of α' martensite in the 14Mn alloy increases with increasing deformation strain; when

the deformation strain is greater than 4%, α' martensite is introduced in the 18Mn alloy; whereas almost no α' martensite is introduced in the 21Mn alloy. In addition, it is worth noting that, in the 18Mn alloy, some stress-induced ε martensite intersects and collides without forming black granular α' martensite, instead appearing white like ε martensite. In the 21Mn alloy, the stress-induced ε martensite at the intersections also appears white. TEM characterization shows that these white phases formed at the intersections of stress-induced ε martensite are ε-martensite twins (Fig. 6).

2.3 Effect of Mn content on the width of stress-induced ε martensite

Figure 7 gives the cumulative frequency distribution of the widths of stress-induced ε martensite in the three alloys with different Mn contents after different amounts of deformation at $M_s + 10$ K. After deformation of 4%, 7%, and 9%, the distribution range of stress-induced ε-martensite widths decreases with increasing Mn content. Moreover, under the same deformation strain, the average width of stress-induced ε martensite also decreases with increasing Mn content (Table 2).

2.4 Effect of Mn content on mechanical behavior

Figure 8 shows the relationship between the nominal yield strength $\sigma_{0.2}$ and temperature for the three alloys with different Mn contents. When the deformation temperature is higher than the maximum temperature M_s^σ for the stress-induced ε-martensitic transformation, the alloy preferentially undergoes plastic ···

(Figure: Fig. 4)

Fig. 4 XRD spectra of solution-treated 14Mn, 18Mn and 21Mn alloys subjected to different deformation strains at their M_s+10 K, and volume fraction of stress-induced ε martensite.

Fig. 4 XRD spectra of solution treated 14Mn (a), 18Mn (b) and 21Mn (c) alloys subjected to different deformation strains at their $M_s + 10$ K and volume fraction of stress-induced ε martensite (d)

(Figure: Fig. 5)

Fig. 5 Saturation magnetizations of solution-treated 14Mn, 18Mn and 21Mn alloys after different amounts of deformation at $M_s + 10$ K.

Fig. 5 Saturation magnetizations of solution treated 14Mn, 18Mn and 21Mn alloys subjected to different deformation strains at their $M_s + 10$ K

(Figure: Fig. 6)

Fig. 6 TEM image and SAED pattern of the solution-treated 21Mn alloy after 9% deformation at $M_s + 10$ K.

Fig. 6 TEM image (a) and SAED pattern of circle in Fig. 6a (b) of 21Mn alloy subjected to 9% deformation at its M_s+10 K (The subscripts T and ε represent

hcp twin and stress-induced ε martensite, respectively)

Deformation[23]. $\sigma_{0.2}$ decreases as the deformation temperature rises. When the deformation temperature is between $M_s + 10$ K and M_s^σ[24], the alloy preferentially undergoes stress-induced ε martensitic transformation, and $\sigma_{0.2}$ increases with increasing deformation temperature. Here, $\sigma_{0.2}$ is the critical stress σ_ε for the stress-induced $\gamma \rightarrow \varepsilon$ martensitic transformation. The austenite yield strength $\sigma_{\gamma\text{slip}}$ of the alloy at $M_s + 10$ K can be obtained by extrapolating the nominal yield strength $\sigma_{0.2}$ at temperatures above M_s^σ, as indicated by the dashed line in Fig. 8. It can be seen from Fig. 8 that, at $M_s + 10$ K, $\sigma_{\gamma\text{slip}}$ increases with increasing Mn content, and the difference $\Delta\sigma_{0.2}$ between $\sigma_{\gamma\text{slip}}$ and σ_ε also increases with ···

(Figure: Fig. 7)

Fig. 7 Cumulative frequency distribution graphs of stress-induced ε martensite width for solution-treated 14Mn, 18Mn, and 21Mn alloys subjected to deformation strains of 4% (a), 7% (b), and 9% (c) at their $M_s + 10$ K

Table 2 Average width of stress-induced ε martensite for solution-treated 14Mn, 18Mn, and 21Mn alloys subjected to different deformation strains at their $M_s +$ 10 K

Alloy	4%	7%	9%
14Mn	0.88	0.91	1.15
18Mn	0.49	0.50	0.85
21Mn	0.38	0.40	0.63

Unit: m

(Figure: Fig. 8)

Fig. 8 Relationships between 0.2% proof stress $\sigma_{0.2}$ and deformation temperatures of solution-treated 14Mn (a), 18Mn (b), and 21Mn (c) alloys (M_s^σ represents the highest temperature of stress-induced ε martensite transformation)

increases with increasing Mn content.

3 Analysis and Discussion

The shape memory effect of Fe-Mn-Si-based alloys originates from the stress-induced $\gamma \leftrightarrow \varepsilon$ martensitic transformation and its reverse transformation

. To obtain a good shape memory effect, the following two conditions must be satisfied: (1) during deformation, the shape change is borne by the stress-induced ε martensitic transformation, avoiding the introduction of plastic slip;

(2) the stress-induced ε martensite has good reversibility

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studied the application of a relatively low stress at 523 K, causing the shape change during cooling of the alloy to be completely induced by stress—

developed ε-martensitic transformation to accommodate it, but its shape memory effect ultimately was not improved; that is, the reversibility of the stress-induced ε-martensite was not guaranteed.

The difference $\Delta\sigma_{0.2}$ between $\sigma_{\gamma\ \mathrm{slip}}$ and σ_{ε} can be used to characterize the ability of Fe-Mn-Si-based shape memory alloys to resist plastic slip. The larger $\Delta\sigma_{0.2}$ is, the less likely plastic slip is to occur in the alloy during deformation. During deformation at $M_s + 10$ K, $\Delta\sigma_{0.2}$ of the Fe-(14–21)Mn-5.5Si-8.5Cr-5Ni alloys increases with increasing Mn content (Fig. 8). This result indicates that the ability of Fe-Mn-Si-Cr-Ni alloys to resist plastic slip increases with increasing Mn content, and therefore the shape memory effect of the alloy also increases with increasing Mn content (Fig. 1). Accordingly, increasing the Mn content helps Fe-Mn-Si-based alloys satisfy condition (1) for obtaining a good shape memory effect.

After the same amount of deformation at $M_s + 10$ K, the shape memory effect of the Fe-(14–21)Mn-5.5Si-8.5Cr-5Ni alloys increases with increasing Mn content, but their volume fraction of stress-induced ε-martensite decreases with increasing Mn content (Figs. 3 and 4); that is, the less stress-induced ε-martensite there is, the better the shape memory effect of the alloy. This means that the reversibility of stress-induced ε-martensite in Fe-Mn-Si-based alloys increases with increasing Mn content. On the one hand, in the three alloys with different Mn contents, the width of stress-induced ε-martensite generated after deformation decreases with increasing Mn content (Fig. 7 and Table 2). The results of Kajiwara[^27] and Bergen et al.[^28] showed that thin, strip-like stress-induced ε-martensite has higher reversibility. Therefore, increasing the Mn content is beneficial to improving the reversibility of stress-induced ε-martensite. On the other hand, in the three alloys with different Mn contents, the amount of α'-martensite introduced at the intersections of stress-induced ε-martensite after deformation at $M_s + 10$ K decreases with increasing Mn content. In the 21Mn alloy, almost no α'-martensite is introduced; instead, ε-martensite kinks are formed (Figs. 3 and 6). When ε-martensite from two variants forms ε-martensite kinks at their intersections, these kinks exert a strong constraining effect on the surrounding ε-martensite, making it difficult for the ε-martensite of the two variants at the intersection to form other phases[^29]. Studies[^30] have also shown that the introduction of α'-martensite in Fe-Mn-Si-based alloys hinders the reverse transformation of stress-induced ε-martensite, thereby degrading the shape memory effect. Therefore, increasing the Mn content suppresses the introduction of α'-martensite and thus improves the reversibility of stress-induced

ε-martensite. In summary, Mn improves the reversibility of stress-induced ε-martensite in Fe-Mn-Si-Cr-Ni alloys from the above two aspects, enabling the alloys to satisfy condition (2) for Fe-Mn-Si-based alloys to obtain a good shape memory effect.

When the deformation temperature is slightly higher than M_s, the stress required for the $\gamma \rightarrow \varepsilon$ transformation is smaller, and less plastic slip is introduced during deformation; therefore, the shape memory effect of Fe-Mn-Si-based alloys is better[^18,^31]. Because M_s decreases with increasing Mn content, during deformation at room temperature the deformation temperature becomes increasingly far from M_s, which causes an increasing portion of the deformation to be borne by plastic slip. Consequently, at this time, the more stress-induced ε-martensite formed in the alloy, the better the shape memory effect. This explains the results of Otsuka et al.[^17] and of Inagaki and Inoue[^18] for alloys with Mn contents between 18% and 28%, namely, the more stress-induced ε-martensite formed, the better the shape memory effect of the alloy. When thermally induced ε-martensite exists in Fe-Mn-Si-based alloys, although the amount of stress-induced ε-martensite formed during deformation increases with increasing Mn content, the reversibility of the stress-induced ε-martensite decreases with increasing Mn content. This explains the results of Inagaki and Inoue[^18] for alloys with Mn contents between 14% and 18%: when thermally induced ε-martensite exists, the more stress-induced ε-martensite there is, the poorer the shape memory effect.

4 Conclusions

(1) When deformed at $M_s + 10$ K, the shape memory effect of the Fe-(14-21)Mn-5.5Si-8.5Cr-5Ni alloys increases with increasing Mn content.

(2) When the Fe-(14-21)Mn-5.5Si-8.5Cr-5Ni alloys are deformed at M_s+10 K, the difference between the yield strength $\sigma_{\gamma\ \mathrm{slip}}$ of austenite and the critical stress σ_ε for stress-induced ε-martensite increases with increasing Mn content; that is, the ability of austenite to resist plastic deformation also increases. This is one reason why increasing the Mn content improves the shape memory effect of Fe-Mn-Si-based alloys.

(3) When the Fe-(14-21)Mn-5.5Si-8.5Cr-5Ni alloys are deformed at M_s+10 K, increasing the Mn content reduces the width of stress-induced ε-martensite and suppresses the introduction of α'-martensite, thereby improving the reversibility of stress-induced ε-martensite. This is another reason why increasing the Mn content improves the shape memory effect of Fe-Mn-Si-based alloys.

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