# Influence of Roll and Solution Treatment Processing on Shape Memory Effect of Fe-14Mn-5Si-9Cr-5Ni Alloy

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The shape memory effect was studied in an Fe-14Mn-5Si-9Cr-5Ni alloy rerolled at 1123 K after hot rolling at 1423 K, followed by solution treatment at different temperatures. It was found that the alloy exhibits a maximum degree of shape recovery in a bending test and a complete recovery tensile strain of 2.2% in samples that were solution heated at 973 K for 600 s and then quenched in water. The rerolled processing at 1123 K after hot rolling at 1423 K and the microstructure under solution treatment state are important for obtaining a good shape memory effect in the alloy.

Keywords microstructure, rolling and solution processing, shape memory alloy, shape memory effect

#### 1. Introduction

It has been shown that Fe-Mn-Si and Fe-Mn-Si-Ni-Cr alloys with low stacking fault energy exhibit the shape memory effect associated with  $\gamma$  face-centered cubic (fcc)  $\leftrightarrow \epsilon$  hexagonal close-packed (hcp) transformation (Ref 1-4). The  $\gamma \leftrightarrow \epsilon$  transformation proceeds by one a/6 <112> Shockley partial dislocation moving reversibly on every second {111} austenite plane. It has been reported that Fe-(~28 to 33) Mn-(~4 to 6) Si alloy has a recovery strain of ~2 to 4% (Ref 5) and Fe-13.7Mn-6Si-8.9Cr-5Ni alloy has a recovery strain of 5% under bending prestrain of 8%, but the fully reversible strain is usually less than 2% (Ref 6). The influence of roll and solution treatment processing on the shape memory effect of these alloys has not been studied. In the present study, the shape memory behavior was examined in a polycrystalline Fe-14Mn-5Si-9Cr-5Ni alloy that was rerolled at 1123 K after hot rolling at 1423 K, followed by solution treatment at different temperatures. The present article concentrates on the yield stress and the microstructure under solution treatment state and its correlation with the shape memory effect.

### 2. Experimental Procedure

The composition of the alloy used in this investigation was Fe-14Mn-5Si-9Cr-5Ni. The alloy was prepared by induction melting in an argon gas atmosphere. The ingots 30 mm by 100 mm by 200 mm were hot rolled at 1423 K into 3 mm plates, and then further rerolled at 1123 K into 0.8 mm plates. The test specimens 0.8 mm by 5 mm by 100 mm for the shape memory effect were machined from the plate parallel to the rolling direction. The specimens were solution heated at different temperatures from 773 to 1173 K for 600 s and then quenched in water.

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The shape memory effect was measured by bending and tensile tests. The specimens were bent into half circular shapes with a radius of 18 mm at room temperature in a bending test. The ends of the specimen were perpendicular to the level line (Fig. 1a, 1b), then it was recovered at 573, 673, and 773 K, respectively. The residual angle,  $\theta$ , was measured (Fig. 1c). The degree of shape recovery was determined by  $\eta_{SME} = (90 - 1)^{-1}$  $\theta$ )/90 × 100%. Tensile deformations were conducted at room temperature on an Instron type machine with a cross head speed of 1 mm/min. The specimens were deformed by various amounts of strain, then unloaded and heated to 673 K for the reversion of stress-induced  $\varepsilon$  martensite. Following cooling, the degree of shape recovery was determined by  $\eta_{SME} = (L_1 - L_2)$  $L_2$ /( $L_1 - L_0$ ) × 100%, where  $L_0$ ,  $L_1$ , and  $L_2$  are the gage length before and after the deformation and after the recovery, respectively. The gage length was measured by using a tool microscope. Values of yield stress was determined from the 0.2% proof stress. Specimens for transmission electron microscopy (TEM) examination were mechanically thinned to 0.1 mm, then electropolished by using the double jet technique in an electrolyte of 20% sulfuric acid and 80% methanol solution at ambient temperature. Transmission electron microscopy observation was performed with a JEM-200 CX electron microscope.

### 3. Results

Figure 2 shows the temperature dependence of the degree of shape recovery in a bending test. It can be seen that the alloy exhibits a maximum shape recovery at the solution heating temperature 973 K, and the degree of shape recovery at the recovery temperature 773 K is higher than that at 573 K.



Fig. 1 Schematic illustration of bending test used for measuring the degree of shape recovery

Figure 3 shows the degree of shape recovery against the tensile prestrain at room temperature for this alloy, which was solution heated at 973 K and 1173 K. It has been shown that a complete recovery strain of 2.2% can be reached after being solution heated at 973 K, but the degree of shape recovery is less than 80% under the same prestrain after being solution heated at 1173 K.



**Fig. 2** Temperature dependence of the degree of shape recovery in Fe-14Mn-5Si-9Cr-5Ni alloy in bending test,  $\Delta$ ,  $\bullet$ , and  $\bigcirc$  at recovery temperatures 573, 673, and 773 K, respectively



Fig. 3 Change in the degree of shape recovery with increasing the tensile prestrain at R, T, ○ solution heated at 973 K. ● solution heated at 1173 K

Figure 4 shows the effect of the solution heating temperature on the yield stress for this alloy after rerolling at 1123 K. The yield stress for this alloy after being rerolled at 1123 K is 560 MPa, and with increasing the solution heating temperatures from 773 to 1073 K, the yield stress results in a significant decrease; it is a minimum at the solution heating temperatures 1073 and 1173 K.

The microstructure of this alloy after being rerolled at 1123 K consists of austenite and  $\varepsilon$  martensite (the dark lath in Fig. 5a), and a high density of the dislocations in the austenite. When the specimens were solution heated to 773 K the  $\varepsilon$  martensite transformated reversely to austenite, and many black bands still remained in the original martensite sites. Figure 5(b) shows the TEM morphology of the black bands, and the electron diffraction pattern confirmed that no  $\varepsilon$  martensite was present. There was evidence under high magnification that such black bands consist of the dislocation tangles. With increasing solution heating temperature, the dislocation tangles in the original  $\varepsilon$  martensite sites scatter. At the solution heating temperature 973 K, the annealing twins form in low dislocation density sites in austenite, and many stacking faults exist in the annealing twins, and the recrystallization is evident in the form of thin plate form in the original  $\varepsilon$  martensite sites (bright plate in Fig. 5c). At solution temperatures above 1073 K, recrystallization is accomplished, dislocations in austenite are removed by the recrystallization, and only some stacking faults and a small amount of thermally induced  $\varepsilon$  martensites exist in austenite. Fig. 5(d) is the TEM morphology of this alloy, which was solution heated at 1173 K and then quenched in water.

### 4. Discussion

The shape memory effect of the Fe-14Mn-5Si-9Cr-5Ni alloy has been studied by different authors (Ref 6-9). This al-



Fig. 4 Plots of yield stress versus the solution heating temperatures

loy was previously prepared by a cold rolling after hot rolling, followed by a solution treatment (Ref 7). Reyhani and McCormick (Ref 8) showed that a maximum shape recovery of this alloy was exhibited by specimens annealed at 1273 K, and the complete recovery strain was less than 1%. In the works of Yang, Chen, and Waymen (Ref 6), although a higher net reversible strain was made available by over prestraining, the fully reversible strain did not exceed 2%. In the present investigation, the results of Fig. 2 and 3 indicate that the maximum degree of shape recovery is associated with the solution heating temperature 973 K in the Fe-14Mn-5Si-9Cr-5Ni alloy, which was rerolled at 1123 K after hot rolling at 1423 K, and the complete recovery of tensile strain of 2.2% is obtained by using the present processing. It seems that a rerolling step at a lower temperature after hot rolling at 1423 K is important for obtaining a good shape memory effect.

Although the yield stress of parent phase after rerolling at 1123 K or solution heated at 773 and 873 K is fairly high (Fig. 4), deformation hardening, high density of dislocations in austenite (Fig. 5a), and dislocation tangles in the original  $\varepsilon$  martensite sites (Fig. 5b) will lead to the difficulty of the a/2

 $[\overline{101}] \rightarrow a/6 \ [\overline{211}] + a/6 \ [\overline{112}]$  dislocation reaction. At the higher solution heating temperatures above 1073 K, the accomplishment of the recrystallization results in low density of dislocations in austenite (Fig. 5d) and low critical resolved shear stress  $\tau_{110}$  because of the minimum yield strength of the alloy (Fig. 4). In this case, the strain induced  $\gamma \rightarrow \varepsilon$  martensite transformation is accompanied by the motion of a usual a/2 < 110 >dislocation so the irreversible plastic deformation also results in a low degree of shape recovery. At the solution heating temperature 973 K, recrystallization occurs in the form of thin plates that formed in the original  $\varepsilon$  martensite sites because the high crystal deformation energy can divide the austenite grain into small districts (Fig. 5c), which can lead to uniform stress distribution and single oriented  $\varepsilon$  martensites during prestraining. It has also been found that the annealing twin formed at the solution heating temperature 973 K, has many stacking faults that can contribute to the nucleation for the stress induced  $\epsilon$ martensite. This is the reason why the maximum degree of shape recovery in the bending test and a complete recovery tensile strain of 2.2% can be obtained at the solution heating temperature 973 K.



**Fig. 5** Transmission electron microscopy morphology of an Fe-14Mn-5Si-9Cr-5Ni alloy after rerolling at 1123 K and solution treatment at different temperatures. (a) After rerolled at 1123 K. (b) Solution treated at 773 K, then quenched in water. (c) Solution treated at 973 K, then quenched in water. (d) Solution heated at 1173 K, then quenched in water

## 5. Conclusions

- When the Fe-14Mn-5Si-9Cr-5Ni alloy was rerolled at 1123 K after being hot rolled at 1423 K, a maximum degree of shape recovery in the bending test and complete recovery tensile strain of 2.2% were present in samples that had been previously solution heated at 973 K and then quenched in water.
- At the solution heating temperature 973 K, the recrystallization in the form of thin plates annealing twins and the stacking faults in austenite are profitable for obtaining the larger degree of shape recovery and complete recovery strain.

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