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## CHARACTERIZATION OF INTERNAL FRICTION OF Fe-30Mn-6Si-5Cr

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### Introduction

Fe-Mn-Si iron-based shape memory alloys have attracted much attention due to their low cost and excellent workability. Many papers [1–4] reported that Fe-Mn-Si alloys, containing 28 to 34 wt% Mn and 4 to 6.5 wt% Si, exhibit a nearly perfect shape memory effect (SME). Moreover, Cr and Ni have recently been added to the Fe-Mn-Si alloys resulting in an improved SME and corrosion resistance [5,6]. Unlike TiNi and Cu-based shape memory alloys, Fe-Mn-Si alloys exhibit a non-thermoelastic martensitic transformation. Their SME comes from the reverse transformation of stress-induced  $\epsilon$ -martensite (HCP structure) into  $\gamma$ -parent austenite (FCC structure) on heating [1]. In the past decade, extensive studies for the Fe-Mn-Si alloys have been conducted on the transformation behaviors [1,7–9], physical properties [7–10] and composition dependence of SME and corrosion resistance [5,11–13]. Much effort has also been devoted to their practical applications, especially on the “heat-to-shrink” pipe coupling [14]. More recently, thermo-mechanical training, in which the alloys are treated by the repetition of small amounts of tensile deformation at room temperature, followed by subsequent annealing at 500~600°C, has been reported to improve effectively the SME of Fe-Mn-Si alloys [15–17]. To extend their applications, a better understanding of Fe-Mn-Si shape memory alloys is necessary, including their shape memory characteristics, phase transformation temperatures and thermo-mechanical treatments. Owing to its high sensitivity on constitutional structures, internal friction measurement has been used to investigate the martensitic transformations for thermoelastic alloys [18,19] and ferrous alloys [20,21]. In our previous paper [7], the internal friction characteristics due to  $\gamma \leftrightarrow \epsilon$  martensitic transformation have been examined by simple heating and cooling cycles. In the present study, the characterization of internal friction with the change in deformation states on an Fe-30Mn-6Si-5Cr shape memory alloy is investigated. In addition, the effect of thermo-mechanical training on the SME of this alloy is also discussed.

### Experimental Procedure

The vacuum melting technique was employed to prepare the Fe-30Mn-6Si-5Cr (wt%) alloy. The as-cast ingot was homogenized at 1100°C for 24 hours and then hot-rolled into 5-mm thickness by using a

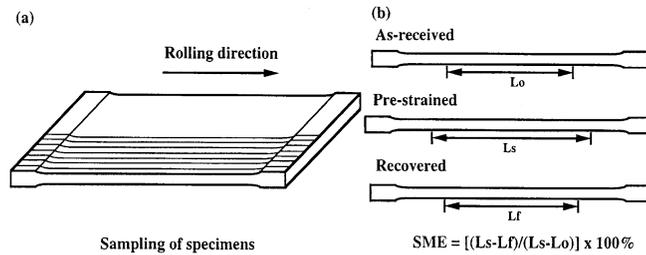


Figure 1. Schematic diagrams illustrate (a) sampling method and (b) the evaluation of shape memory effect.

two-high hot-rolling mill. Specimens for SME, internal friction, X-ray diffraction and tensile test were carefully machined from this hot-rolled plate. The SME was examined by a tensile test. Specimens were tensile-strained with a variety of strain and then reheated to 500°C for 10 min. The related SME was then calculated. Figure 1 illustrates the sampling method and the evaluation of SME. To determine the effect of thermo-mechanical training on the SME, specimens were treated by the repetition of a 5% tensile strain and subsequent heating up to 500°C for several times. The internal friction test was carried out using a SINKU-RIKO 1500M/L series inverted torsion pendulum in the temperature range from -150°C to 500°C. The measuring frequency was about 1 Hz, and the heating and cooling rate was kept at 2°C/min. The recording of data was completely automatic. A digit computer carried out the calculation and plotted the curve of internal friction  $Q^{-1}$  versus temperature. Thus, the results obtained have a rather good resolution. X-ray diffraction analysis was performed at room temperature with the Siemens D500 X-ray diffractometer using  $MoK\alpha$  radiation. Meanwhile, a Shimadzu tensile tester was applied to measure the mechanical properties of tested specimens.

## Results and Discussion

Table 1 presents the results of SME test for the specimens with a variety of pre-strain. As shown in the Table 1, the 2% pre-strained specimen can exhibit a nearly perfect SME. Nevertheless, the SME decreases rapidly with increasing pre-strain applied. This feature indicates that the Fe-30Mn-6Si-5Cr alloy could be favored for application only under a small strain condition, say 5% for the as hot-rolled specimen. As mentioned above, the thermo-mechanical training can improve effectively the SME of Fe-Mn-Si alloys. This phenomenon is also confirmed in this study. Table 2 presents the effect of thermo-mechanical training on the SME for the 5% pre-strained condition. As can be seen in Table 2, the SME is considerably improved from 75% up to 86%, along with the cyclic training number increased from the first to the 4th cycle.

Figure 2 shows the plots of internal friction  $Q^{-1}$  versus temperature for the Fe-30Mn-6Si-5Cr alloy of the as hot-rolled as well as 2%, 5% and 10% pre-strained specimens. In Fig. 2, there is a sharp internal friction peak at around 120°C for the as hot-rolled specimen. For the 2% pre-strained specimen, the internal friction peak at 120°C is broadened. By increasing the pre-strain to 5%, a very wide internal friction spectrum with a peak temperature of 200°C can be observed. Furthermore, for the 10%

TABLE 1  
Effect of the Pre-Strain on the SME

Pre-strained %	2	5	10	12	15
SME %	99	75	39	29	18

TABLE 2  
Effect of Thermo-Mechanical Training on the SME for 5% Pre-Strained Condition

Cyclic training number	1	2	3	4
SME %	75	77	82	86

pre-strained specimen, there is another obvious internal friction peak appearing at 300°C. Under a careful analysis, these internal friction spectra could be classified into three peaks which locate at around 120°C, 200°C and 300°C. The wide internal friction spectrum of the 5% pre-strained specimen just comes from the combination of these three peaks. Throughout the decomposition of the internal friction spectrum, the  $Q^{-1}$  values of 120°C, 200°C and 300°C peaks are found to be  $6.2 \times 10^{-3}$ ,  $9.1 \times 10^{-3}$  and  $4.6 \times 10^{-3}$ , respectively. The effect of thermo-mechanical training on the internal friction for the 5% pre-strained condition is also illustrated in Fig. 3. As can be seen in Fig. 3, the maximum height of the 200°C peak increases with increasing cyclic training number.

To analyze and explain the related features and physical meaning of the internal friction spectra observed in Figs. 2 and 3, X-ray diffraction was carried out on both the as hot-rolled and 5% pre-strained specimens. Figure 4 (a) and (b) show the X-ray diffraction spectra of the as hot-rolled and 5% pre-strained specimens, respectively. It reveals that the stress-induced  $\epsilon$ -martensite is present in the pre-strained specimen, but not in the as hot-rolled specimen. Hence, the 120°C peak found in the as hot-rolled specimen is ascribed to the reverse transformation of the thermal-induced  $\alpha'$ -martensite (BCT structure) into the parent austenite phase. On the other hand, the 200°C peak could be attributed to the reverse transformation from the stress-induced  $\epsilon$ -martensite to the parent austenite phase. As shown in Fig. 3, the maximum height of the 200°C peak increases with increasing cyclic training number. Two reasons may account for this feature. First, the volume fraction of the stress-induced  $\epsilon$ -martensite is increased, and second, the  $\epsilon$ -martensite becomes easily reverse transformed into the parent austenite phase after thermo-mechanical training. Both of the above conditions will also improve the SME. Jiang et al. [16,17] reported that, with the aid of autocatalytic activation mechanism, much more preferentially oriented and refined  $\epsilon$ -martensite plates would be generated after cyclic thermo-mechanical training. On heating, these preferentially oriented and refined  $\epsilon$ -martensite plates can easily

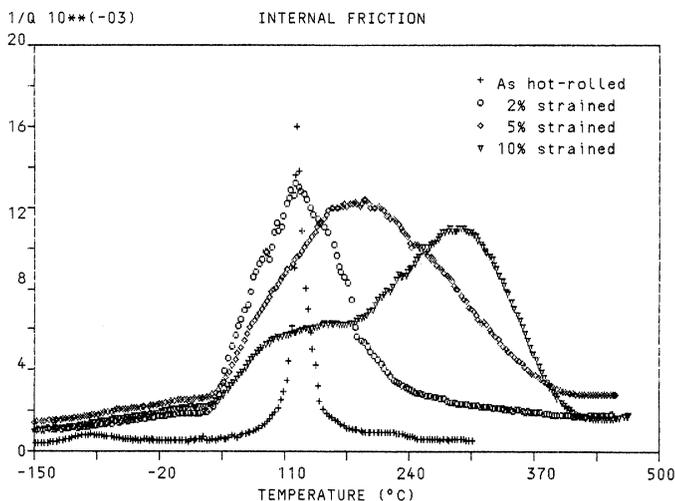


Figure 2. Internal friction of the as hot-rolled and different pre-strained Fe-30Mn-6Si-5Cr specimens.

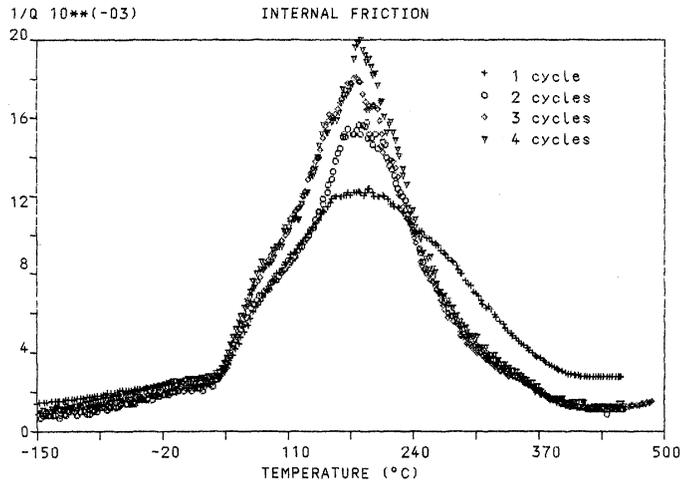


Figure 3. Internal friction of the cyclic thermo-mechanical training Fe-30Mn-6Si-5Cr specimen.

reverse transform into the parent austenite phase. Fig. 3 also indicates that the maximum height of the 200°C peak is increased while its peak temperature is slightly reduced after more cyclic thermo-mechanical training. These features are in good agreement with the fact that a higher volume fraction of the preferentially oriented and refined  $\epsilon$ -martensite, which can easily reverse transform into the parent austenite phase, has been generated after more cyclic thermo-mechanical training. Therefore, the SME is improved, as shown in Table 2.

As seen in Fig. 2, a remarkable internal friction peak appears at around 300°C for the 10% pre-strained specimen. The higher the pre-strain, the more obvious the 300°C peak is. Since the dislocation density increases with increasing plastic deformation, the 300°C peak is easily ascribed to the dislocation relaxation occurring in the deformed specimen. For confirmation, the internal friction test was further made on a heavily cold-rolled specimen with a 28% thickness reduction, and the result is shown in Fig. 5. Carefully examining the internal friction spectra shown in Figs. 2 and 5, we can easily find that the maximum height of the 300°C peak increases with increasing plastic deformation,

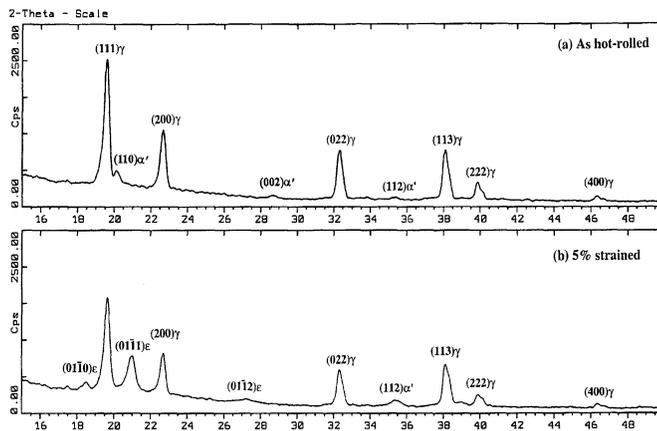


Figure 4. The X-ray diffraction (XRD) spectra of the as hot-rolled and 5% pre-strained samples.

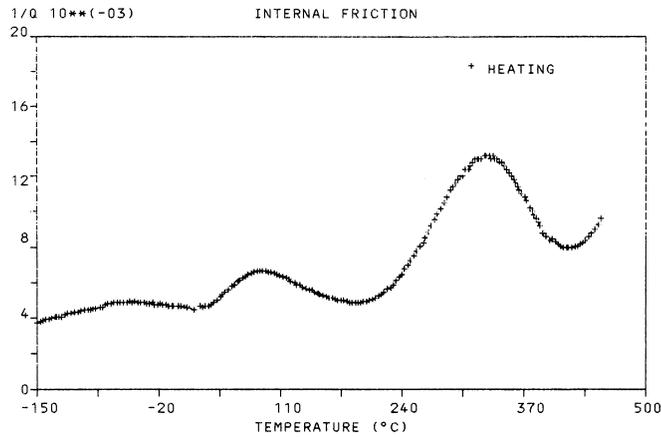


Figure 5. Internal friction of a 28% heavily deformed Fe-30Mn-6Si-5Cr specimen.

whilst the peak temperature for the 28% cold-rolled specimen is raised in comparison with that for the 10% pre-strained specimen. These give evidence that dislocation relaxation results in the 300°C internal friction peak. In addition, the 200°C internal friction peak is not found in the 28% cold-rolled specimen. The absence of the 200°C peak indicates that there is no reverse transformation from the stress-induced  $\epsilon$ -martensite into the parent austenite phase. This can elucidate why the 28% cold-rolled specimen exhibits no SME character even though it has been reheated to 600°C.

By applying thermo-mechanical training, the 300°C dislocation-relaxation peak is weakened. This can be easily observed from the variation in  $Q^{-1}$  value on the right shoulder of the internal friction spectra shown in Fig. 3, in which the  $Q^{-1}$  value of 300°C peak is changed from  $4.6 \times 10^{-3}$  for the first cycle to  $1.8 \times 10^{-3}$  for the 4th cycle. It indicates that a large quantity of dislocations could occur in the austenite matrix during the pre-strain deformation. During thermo-mechanical training up to 500°C, the dislocations would be annealed and hence the 300°C peak is weakened. Table 3 displays the yield strength and work hardening exponent of the specimen having different training cycles of 5% pre-strain. It shows that the yield strength decreases, and the work hardening exponent increases with increasing training cycles. This indicates that the dislocation density is indeed decreased throughout the thermo-mechanical training. According to the above discussion, thermo-mechanical training can enhance the generation of refined  $\epsilon$ -martensite and decrease the dislocation density in the parent austenite phase, thus improving the SME.

### Conclusions

The characterization of internal friction and related SME of an Fe-30Mn-6Si-5Cr alloy has been investigated. The internal friction spectra are in fact classified into three peaks, which locate at around

TABLE 3  
Effect of Thermo-mechanical Training on the Tensile Properties of Fe-30Mn-6Si-5Cr Alloy

Cyclic training number	1	2	3	4
Yield strength, MPa	410.6	377.2	371.9	370.2
$n_{2-4}$	0.229	0.256	0.273	0.271

Yield strength: 0.2% offset strength

$n_{2-4}$ : work hardening exponent under the strain ranging from 2 to 4%

120°C, 200°C and 300°C. The 120°C and 200°C peaks are ascribed to the reverse transformation of  $\alpha'$ -martensite and stress-induced  $\epsilon$ -martensite, respectively, into the parent austenite phase. The 300°C peak is attributed to the dislocation relaxation found in the austenite matrix. With increasing pre-strain, the maximum height of the 200°C peak decreases while that of the 300°C peak is sharpened. It reveals that the SME will decrease with increasing pre-strain. Meanwhile, the maximum height of the 200°C peak is increased while the peak temperature is decreased after cyclic thermo-mechanical training. These features are in good agreement with the fact that a higher volume fraction of preferentially oriented and refined  $\epsilon$ -martensite, which can easily reverse transform into the parent austenite phase, has been generated after cyclic thermo-mechanical training. Therefore, the SME is improved.

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