



# Role of thermal martensite in shape memory effect of CoAl and CoNi alloys

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**Abstract:** To address the role of the HCP martensite in CoAl and CoNi shape memory alloys, the relationship between the shape memory effect (SME) and the content of the thermal and stress-induced HCP martensite was investigated in the solution-treated CoAl and CoNi alloys. In-situ optical observations were employed to investigate the contents of thermal HCP martensite before and after deep cooling and its influence on the stress-induced HCP martensite transformation and SME. The results show that the SME in both the CoAl and the CoNi alloys results from the stress-induced HCP martensite. The role of the thermal HCP martensite in both of them is the strengthening of the matrix. The much higher yield strength in the solution-treated CoAl alloy due to solution strengthening of Al is responsible for its better SME compared with the CoNi alloy.

**Key words:** CoAl alloy; CoNi alloy; shape memory effect; thermal HCP martensite; stress-induced HCP martensite; solution strengthening

## 1 Introduction

CoAl alloys undergoing a face-centered cubic (FCC)  $\rightleftharpoons$  hexagonal close packed (HCP) martensitic transformation have drawn much attention in the past decade as a new type of shape memory alloy (SMA) [1–5]. On the one hand, both the FCC and HCP phases in the CoAl alloys are ferromagnetic, but in contrast, they are both paramagnetic in the Fe–Mn–Si-based alloys showing a good shape memory effect (SME) due to stress-induced FCC  $\rightarrow$  HCP martensite transformation and its reverse transformation [6–12]. On the other hand, the start temperature of reverse transformation of HCP martensite in the CoAl alloys can be above 473 K, depending on the Al content [3]. Therefore, they are considered as attractive candidates for high-temperature and ferromagnetic SMAs. However, the SME in the CoAl alloys is much poorer than that in the Fe–Mn–Si-based alloys. Besides, the relationship between the pre-existing thermal HCP martensite and the SME is still unclear in the CoAl SMAs so far, although it is generally thought that the pre-existing thermal HCP martensite deteriorates the SME in the Fe–Mn–Si-based alloys [9].

ANDO et al [4] pointed out that the SME increased with the rise in the content of thermal HCP martensite in

the  $(\text{Co}_{90}\text{Al}_{10})_{100-y}\text{X}_y$  ( $\text{X}=\text{Si}, \text{Ti}, \text{V}, \text{Cr}, \text{Mn}, \text{Fe}, \text{Ni}, \text{Nb}, \text{Mo}, \text{Ta}$  or  $\text{W}$ ) ternary alloys. After being bent by 1.2%, the SME in the alloys containing above 95% thermal HCP martensite could reach 70%, but it dropped sharply to 20% in the alloys without thermal HCP martensite due to more addition of X elements. Based on these results, they suggested that a high fraction of the HCP martensite phase was probably one of the reasons for excellent SME in the CoAl binary alloys. However, they did not investigate why a high volume fraction of HCP martensite phase tended to cause a good SME in the CoAl alloys. In addition, the relationship between the stress-induced HCP martensitic transformation and SME in the CoAl alloy has not been reported up to date.

The same phenomenon, that is, the SME rises with increasing the content of thermal HCP martensite, exists in the CoNi binary alloys which also undergo the FCC  $\rightleftharpoons$  HCP martensitic transformation [13–15]. SHIN et al [13] thought that the SME in the CoNi alloys resulted from the coalescence of pre-existing thermal HCP martensite, not the stress-induced FCC  $\rightarrow$  HCP martensite transformation and its reverse transformation. However, ZHOU et al [16] suggested that the SME in the CoNi alloys resulted also from the stress-induced HCP martensite, as the Fe–Mn–Si-based SMAs do. Very recently, SUN et al [15] have clarified that the origin of the SME in the CoNi alloys still resulted from the

stress-induced HCP martensite and its reverse transformation. The role of the thermal HCP martensite was strengthening of matrix. The more the pre-existing thermal HCP martensite is, the greater the strengthening of matrix is, and the more the stress-induced HCP martensite is. Accordingly, the SME increases with the rise in the content of thermal HCP martensite.

Based on the above results and discussion, a question, whether the SME in the CoAl alloys also originates from the stress-induced HCP martensite and its reverse transformation, will arise. Is the role of thermal HCP martensite also strengthening of the matrix, as it is in the CoNi alloys? To answer these questions, in this work, the evolution of the content of thermal HCP martensite before and after deep cooling at 77 K and its influence on the stress-induced HCP martensite transformation and SME were investigated in a CoAl alloy and a CoNi alloy using in-situ color optical characterization, respectively.

## 2 Experimental

The CoAl and CoNi alloys were prepared by induction melting under a vacuum atmosphere, using high pure cobalt, aluminum and nickel. The ingots were hot-rolled into sheet of 2.3 mm in thickness at 1473 K, and then solution-treated at 1473 K for 40 min. Finally, the solution-treated sheets were cold-rolled with a thickness reduction of 10%. Their chemical compositions were Co<sub>2.19</sub>Al and Co<sub>30.30</sub>Ni alloys (mass fraction, %).

The SME was examined by conventional bending techniques [7]. The specimens of 70 mm × 2.0 mm × 1.0 mm cut from cold-rolled sheet were firstly solution-treated at 1373 K for 30 min, followed by air-cooling to room temperature (RT). Then, half of the specimens were soaked in liquid nitrogen for 5 min. Finally, all the specimens undergoing two kinds of heat treatment were bent around a mould to 180° at room temperature, as shown in Fig. 1. The pre-strain  $\varepsilon$  was taken as the maximum tensile strain at the outer edge of the sample thickness and is determined by the equation:  $\varepsilon = h/(2R_d + h)$ , where  $R_d$  is the bend radius and  $h$  is the sample thickness. The bent specimens were then annealed at 973 K for 5 min to recover the deformed shape. After recovery annealing, the residual strain  $\varepsilon_r$  was determined by the equation:  $\varepsilon_r = h/(2R_r + h)$ , where  $R_r$  is the bend radius after recovery. The shape recovery rate ( $\eta_{SRR}$ ) was calculated by the equation:  $\eta_{SRR} = (\varepsilon - \varepsilon_r)/\varepsilon \times 100\%$ . Each presented value was the average value of three specimens. To determine the 0.2% proof yield stress  $\sigma_{0.2}$  at different temperatures, the dog-bone-shaped tensile specimens with gage section dimensions of 70.0 mm × 5.0 mm × 2.0 mm were cut from the cold-rolled sheet using a Mo filament cutter. After they were subjected to the same

two kinds of heat treatment, they were tensile-deformed by 2.4% at different temperatures with a strain rate of 0.1 mm/min using a RGM-4300 universal tensile test machine, respectively. The  $\sigma_{0.2}$  values at different temperatures were determined from the tensile stress-strain curves.

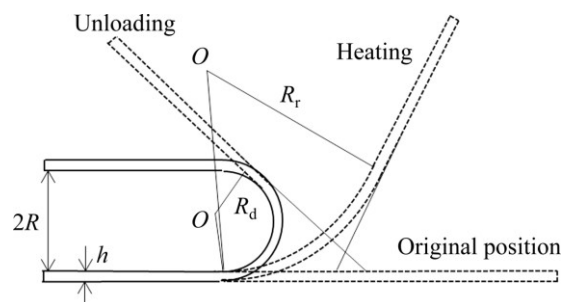


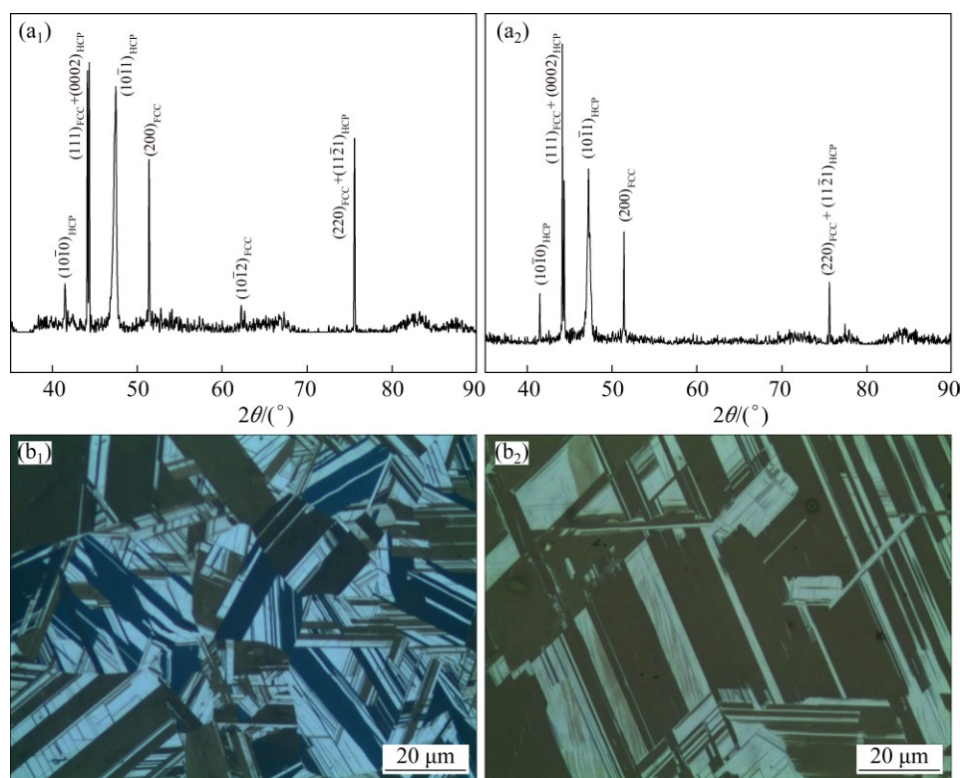
Fig. 1 Schematic diagram showing process of measuring SME

Philips X' Pert Pro MPD X-ray diffraction (XRD) apparatus was used to identify the phases of the specimens. The color optical observation (bright field) was made using OLYMPUS GX51 optical microscope. The specimens before deformation were electro-polished in a solution of 10% HClO<sub>4</sub>+90% C<sub>2</sub>H<sub>5</sub>OH after mechanically polished, and then etched in a solution composed of 1.2% K<sub>2</sub>S<sub>2</sub>O<sub>5</sub> in water. In the color optical image, FCC parent phase appears white, and HCP martensite appears black [15]. After observation before deformation, the specimens were slightly electro-polished to remove the corrosion layer. After deformation, the deformed specimens were not subjected to polishing and directly etched to avoid the effect of preparation of specimens. As the content of the stress-induced HCP martensite was rather low, the content of stress-induced HCP martensite was statistically determined by the image processing soft based on 30 optical micrographs [15].

## 3 Results

### 3.1 Content of thermal HCP martensite and SME in solution-treated alloys

Figure 2(a) shows the XRD patterns of the solution-treated Co<sub>2.19</sub>Al and Co<sub>30.30</sub>Ni alloys at room temperature (298 K), respectively. Several very strong peaks of HCP martensite appeared in both alloys, revealing that lots of thermal HCP martensite existed. Optical micrographs also confirmed the pre-existence of many thermal HCP martensites (black area in Fig. 2(b)). Based on a statistical analysis of 30 optical micrographs, the contents of thermal HCP martensite of Co<sub>2.19</sub>Al and Co<sub>30.30</sub>Ni alloys were determined as 59.1% and 58.7%, respectively.



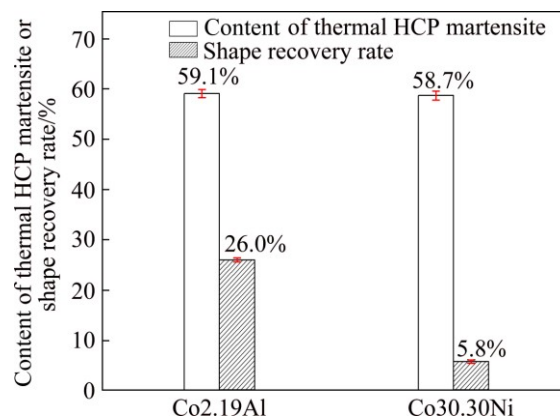
**Fig. 2** XRD patterns ( $a_1$ ,  $a_2$ ) and optical micrographs ( $b_1$ ,  $b_2$ ) of solution-treated Co<sub>2.19</sub>Al ( $a_1$ ,  $b_1$ ) and Co<sub>30.30</sub>Ni ( $a_2$ ,  $b_2$ ) alloys, respectively

The shape recovery rates of the solution-treated Co<sub>2.19</sub>Al and Co<sub>30.30</sub>Ni alloys after being bent by 2.4% at 298 K were 26.0% and 5.8%, respectively. Figure 3 gives their shape recovery rates together with their thermal HCP martensite contents. It was very clear that although their thermal HCP martensite contents were almost the same, the shape recovery rate of Co<sub>2.19</sub>Al alloy was much higher than that of Co<sub>30.30</sub>Ni alloy.

### 3.2 Content of thermal HCP martensite and SME in deep-cooling alloys

Figure 4 shows the optical microstructural evolution at 298 K for the same samples of the solution-treated alloys before and after soaked in the liquid nitrogen for 5 min. The change in microstructure cannot almost be identified in the Co<sub>2.19</sub>Al alloy after being deep-cooled. In contrast, some pre-existing HCP martensite plates (black) obviously grew in size, as indicated by the white arrows. In addition, some new martensite plates (black) were produced in some areas (white squares).

The statistical content of thermal HCP martensite based on 30 optical micrographs clearly reflected this difference in the evolution of martensite content (Fig. 5). The content of thermal HCP martensite in the Co<sub>30.30</sub>Ni alloys remarkably increased to 69.9% after being deep-cooled, but it was 58.7% in the Co<sub>2.19</sub>Al alloys, being almost equal to that before deep-cooling (59.1%). Figure 5 also shows the shape recovery rates of the

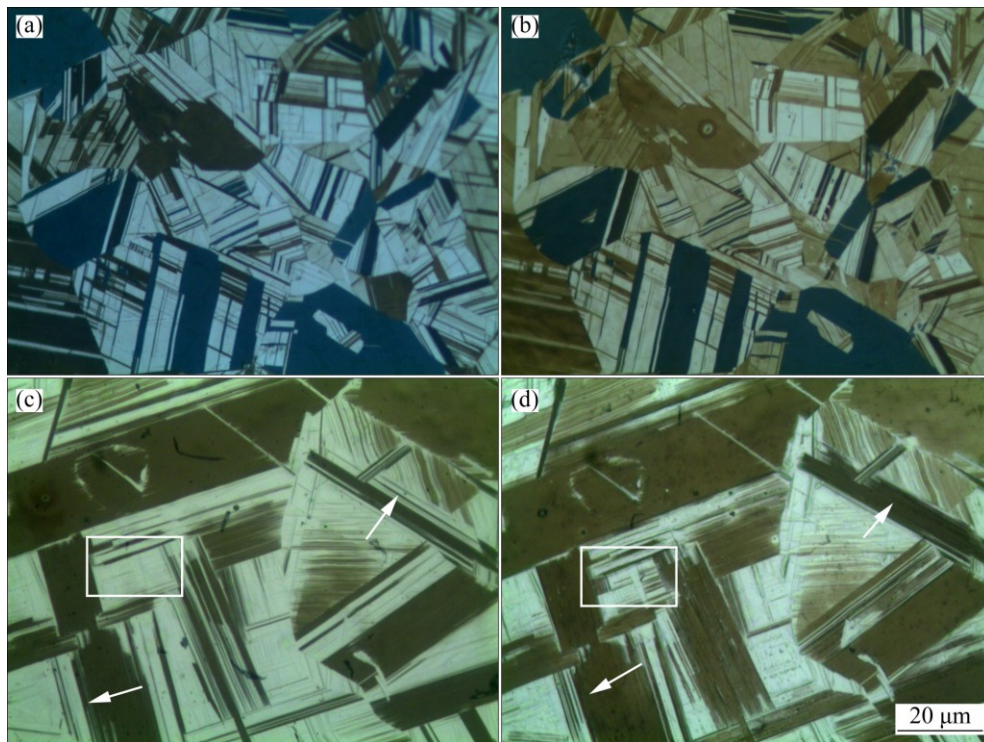


**Fig. 3** Content of thermal HCP martensite and shape recovery rate after being bent by 2.4% at 298 K in solution-treated Co<sub>2.19</sub>Al and Co<sub>30.30</sub>Ni alloys

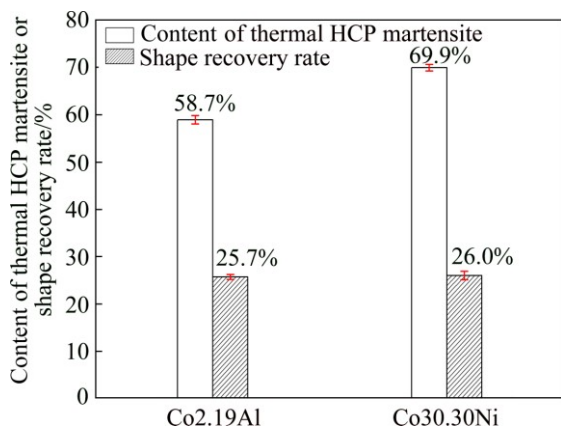
deep-cooled alloys after being bent by 2.4% at 298 K. After being deep-cooled, the  $\eta_{SRR}$  value of the Co<sub>30.30</sub>Ni alloy increased from 5.8% to 26.0%, but the  $\eta_{SRR}$  value of the Co<sub>2.19</sub>Al alloy changed little (25.7%), which was almost the same as that of the solution-treated alloy (26.0%).

### 3.3 Relationship between SME and content of stress-induced HCP martensite

Figure 6 shows the in-situ optical micrographs of the solution-treated alloys before and after 2.4% tensile



**Fig. 4** In-situ optical micrographs of solution-treated Co2.19Al and Co30.30Ni alloys before and after being cooled at 77 K for 5 min: (a) Co2.19Al alloy, before deep-cooling; (b) Co2.19Al alloy, after deep-cooling; (c) Co30.30Ni alloy, before deep-cooling; (d) Co30.30Ni alloy, after deep-cooling



**Fig. 5** Content of thermal HCP martensite and shape recovery rate after being bent by 2.4% at 298 K in solution-treated Co2.19Al and Co30.30Ni alloy subjected to deep cooling in liquid nitrogen for 5 min

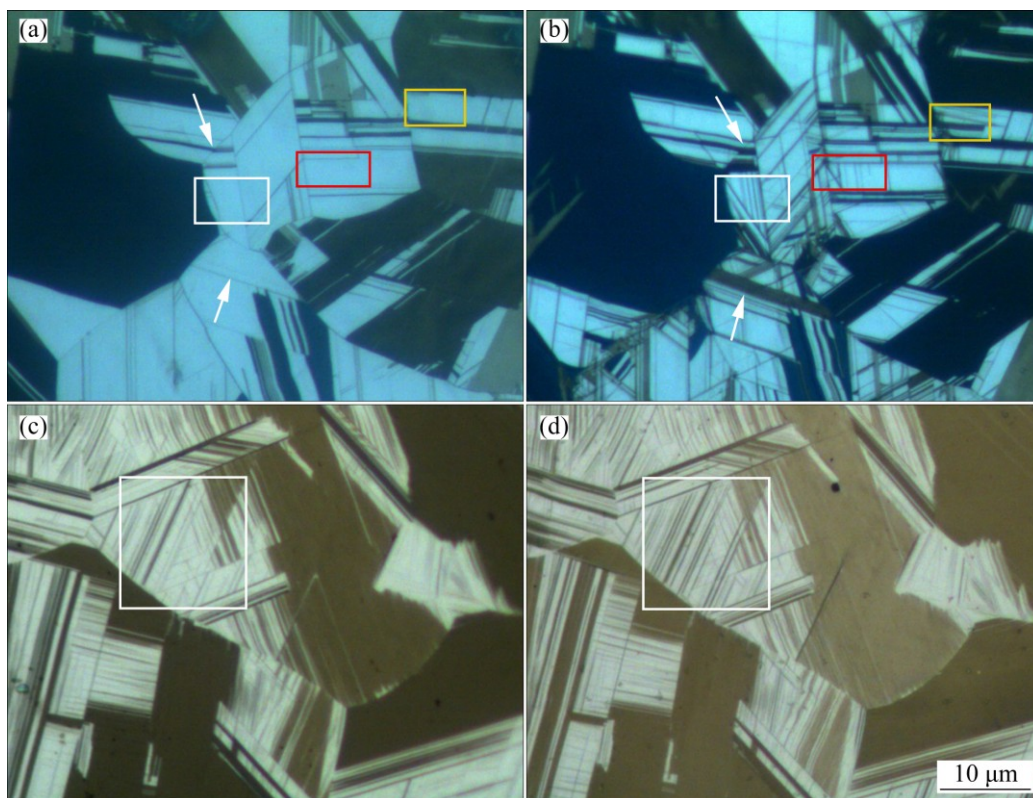
deformation at 298 K, respectively. After deformation, some thin stress-induced martensite plates were produced in both alloys (as shown in the squares). In addition, it can be clearly seen that some pre-existing thermal martensite plates grew in the Co2.19Al alloy (as indicated by the arrows), but they can hardly be seen in the Co30.30Ni alloy. For the deep-cooled alloys, the similar microstructural changes were observed (Fig. 7). Based on 30 optical micrographs, the content of stress-induced HCP martensite after deformation, that is, the difference between the content of thermal HCP

martensite before deformation and that of total HCP martensite after deformation, is given in Fig. 8, respectively. The content of the stress-induced HCP martensite was obviously higher in the solution-treated Co2.19Al alloy than in the solution-treated Co30.30Ni alloy. However, the content of stress-induced HCP martensite in the deep-cooled Co30.30Ni alloy remarkably increased from 1.8% to 4.2%, but it changed a little in the deep-cooled Co2.19Al alloy. To compare the relationship between the content of stress-induced HCP martensite and the SME, Fig. 8 also gives the shape recovery rate. It was very clear that the shape recovery rate showed a positive dependence on the content of stress-induced HCP martensite in both alloys, regardless of the thermal treatment.

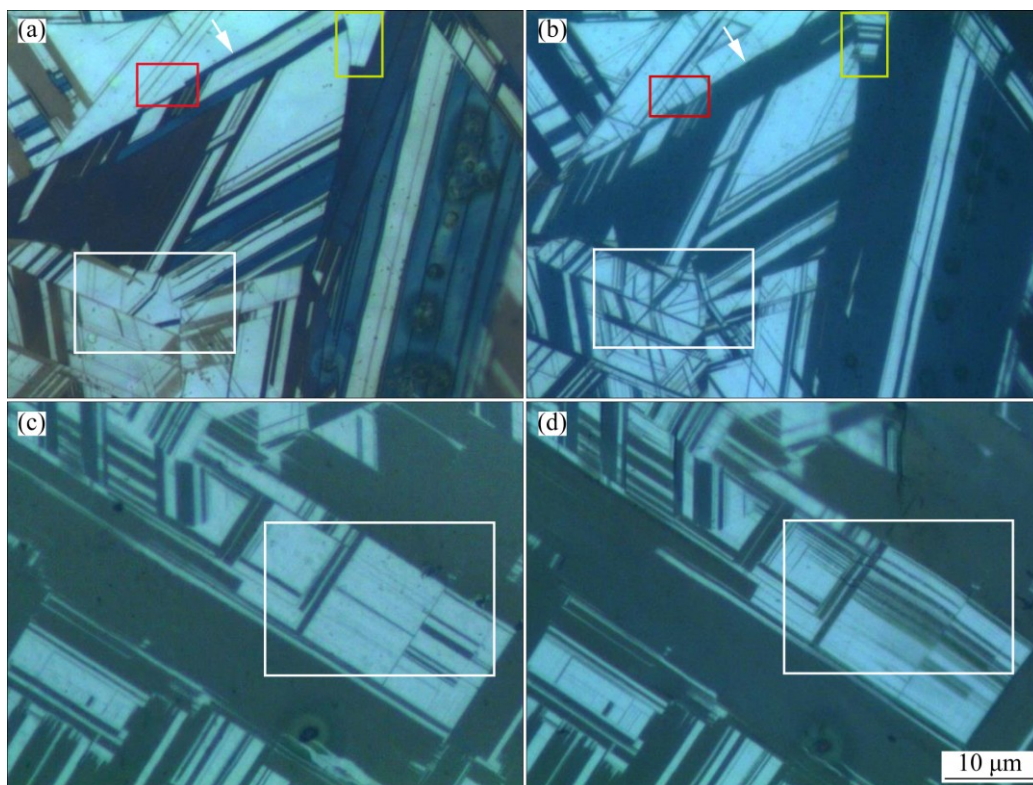
### 3.4 Effect of pre-existing thermal HCP martensite on mechanical behavior

Figure 9 shows the 0.2% proof stress  $\sigma_{0.2}$  of the solution-treated Co2.19Al and Co30.30Ni alloys before and after deep-cooling as a function of temperature. For the solution-treated Co30.30Ni alloy, only a negative temperature dependence of  $\sigma_{0.2}$  existed, that is, the  $\sigma_{0.2}$  increased gradually with lowering the temperature. Note that when temperature was below 298 K, it increased rapidly. In contrast, for the solution-treated Co2.19Al alloy, an obvious positive temperature dependence of  $\sigma_{0.2}$  existed in the region between 298 and 333 K. In addition,

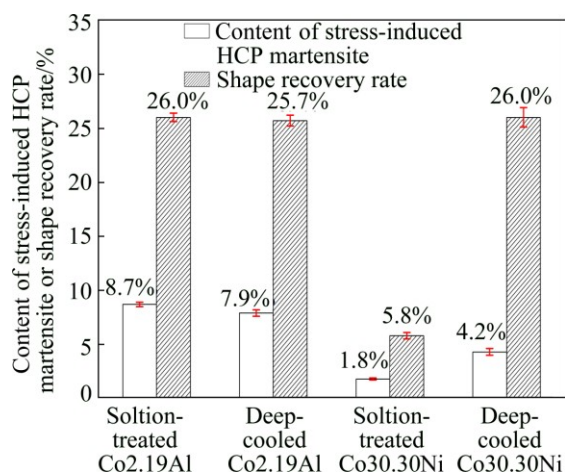




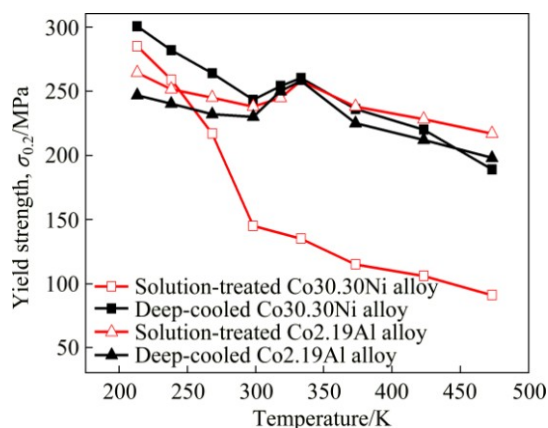
**Fig. 6** In-situ optical micrographs before and after 2.4% tensile deformation at 298 K for solution-treated Co<sub>2.19</sub>Al and Co<sub>30.30</sub>Ni alloy: (a) Co<sub>2.19</sub>Al alloy, before deformation; (b) Co<sub>2.19</sub>Al alloy, after deformation; (c) Co<sub>30.30</sub>Ni alloy, before deformation; (d) Co<sub>30.30</sub>Ni alloy, after deformation



**Fig. 7** In-situ optical micrographs before and after 2.4% tensile deformation at 298 K for solution-treated Co<sub>2.19</sub>Al and Co<sub>30.30</sub>Ni alloys soaked in liquid nitrogen for 5 min: (a) Co<sub>2.19</sub>Al alloy, before deformation; (b) Co<sub>2.19</sub>Al alloy, after deformation; (c) Co<sub>30.30</sub>Ni alloy, before deformation; (d) Co<sub>30.30</sub>Ni alloy, after deformation



**Fig. 8** Content of stress-induced HCP martensite and shape recovery rate in solution-treated and deep-cooled Co2.19Al and Co30.30Ni alloys



**Fig. 9**  $\sigma_{0.2}$  values of solution-treated Co2.19Al and Co30.30Ni alloys before and after deep-cooling at 77 K as function of temperature

the  $\sigma_{0.2}$  of the Co2.19Al alloy was much greater than that of the Co30.30Ni alloy when the temperature was above 333 K, although both of them dropped with increasing the temperature. Interestingly, after deep-cooling, an obvious positive temperature dependence of the  $\sigma_{0.2}$  in the deep-cooled Co30.30Ni alloy appeared in the region between 298 and 333 K. In addition, the  $\sigma_{0.2}$  of the deep-cooled Co30.30Ni alloy increased impressively. It increased by about 100 MPa at the temperatures above 298 K. When the temperature was above 298 K, the  $\sigma_{0.2}$  of the deep-cooled Co30.30Ni alloy was comparable to that of the solution-treated Co2.19Al alloy, although it was much lower before deep-cooling.

## 4 Discussion

It is beyond doubt that the SME in the Fe–Mn–Si-based alloys results from the stress-induced FCC→HCP martensite transformation and its reverse transformation [8,12,17]. Very recently, the studies by SUN et al [15]

have clarified that the origin of SME in the Co–Ni alloys was the same as that of Fe–Mn–Si-based alloys, that is, it also resulted from the stress-induced HCP martensite and its reverse transformation. The direct evidence was that the SME in the Co30.8Ni2.99Si alloy without pre-existing thermal HCP martensite can reach about 60%, but it was only 20% in the Co30.3Ni alloy containing 57% HCP martensite. Furthermore, the results of YAN et al [18,19] showed that both the training and the ausforming treatments, the effective ways of improving the SME in the Fe–Mn–Si-based alloys, can also effectively improve the SME of the CoNiSi alloy. They thought that the much poor SME in the CoNi alloys can be ascribed to their low yield strength, leading to the intrusion of unrecovered permanent deformation, i.e., dislocations slip. The training and ausforming treatments can remarkably strengthen the matrix of the CoNi alloys, thus effectively improving their SME. The effect of strengthening matrix of thermal HCP martensite can explain why the SME of the CoNi-based alloys increases with the rise in the thermal HCP martensite.

In the present work, although the content of thermal HCP martensite in the solution-treated Co2.19Al alloy is comparable to that in the solution-treated Co30.30Ni alloys, its SME is much better than that of the Co30.30Ni alloy (Fig. 3). Obviously, the content of thermal HCP martensite cannot explain this large difference in the SME. However, the differences in the content of stress-induced HCP martensite and the yield strength can explain this large difference. After deformation by 2.4%, the content of stress-induced HCP martensite was only 1.8% in the solution-treated Co30.30Ni alloy, but it reached 8.7% in the solution-treated Co2.19Al alloy. This large difference in the content of stress-induced HCP martensite is in good agreement with the above large difference in the SME.

On the other hand, although the stress-induced HCP martensite takes place in both the solution-treated Co30.30Ni and Co2.19Al alloys, only the  $\sigma_{0.2}$  of the Co2.19Al alloy shows a positive temperature dependence, a characteristic of the stress-induced martensitic transformation. The much lower yield strength in the Co30.30Ni alloy than in the Co2.19Al alloy (Fig. 9) can explain this phenomenon. Because of much lower yield strength, the formation of stress-induced HCP martensite in the Co30.30Ni alloy is accompanied by usual slip during deformation. The occurrence of usual slip will lead to the disappearance of the characteristic of stress-induced martensitic transformation [10]. Furthermore, the intrusion of usual slip not only decreases the content of the stress-induced HCP martensite but also reduces its reversibility [8]. Consequently, the SME of the solution-treated Co30.30Ni alloy must be poorer than that of the

Co<sub>2</sub>.19Al alloy.

After deep-cooling at 77 K, the yield strength of the solution-treated Co<sub>30</sub>.30Ni alloy increased by 100 MPa due to the intrusion of 10% thermal HCP martensite (Fig. 5 and Fig. 9). The remarkable increase in the yield strength can account for the remarkable rise in the content of stress-induced HCP martensite and its resulting improvement of the SME in the deep-cooled Co<sub>30</sub>.30Ni alloy. Therefore, the SME in the CoAl and CoNi alloys results from the stress-induced HCP martensite and its reverse transformation. The role of thermal HCP martensite in CoAl alloys was also strengthening of matrix, as it does in the CoNi alloy [15]. The larger atomic size difference between Al and Co atoms and its greater strengthening effect can explain higher yield strength in the Co<sub>2</sub>.19Al alloy. The decrease in the yield strength in the ternary CoAlX alloys should be the reason for that their SME decreased with the drop in the content of thermal HCP martensite. In the future, it is of great importance to investigate the effect of strengthening matrix on the stress-induced HCP martensitic transformation and the SME in the CoAl alloys.

However, although the content of stress-induced HCP martensite in the deep-cooled Co<sub>30</sub>.30Ni alloy (4.2%) is significantly lower than those of the solution-treated and deep-cooled Co<sub>2</sub>.19Al alloy (8.7% and 7.9%, respectively), its SME is comparable to that of Co<sub>2</sub>.19Al alloy. Very recently, this phenomenon has also been reported in the Fe–Mn–Si-based SMAs [20]. After being deformed by 4% at  $M_s+10$  K, the content of the stress-induced HCP martensite in the solution-treated Fe–14Mn–5.5Si–8.5Cr–5Ni was 21.2%, 5.1% higher than that in the solution-treated Fe–21Mn–5.5Si–8.5Cr–5Ni alloy. On the contrary, the SME in the Fe–14Mn–5.5Si–8.5Cr–5Ni alloy was 8% lower than that of the Fe–21Mn–5.5Si–8.5Cr–5Ni alloy. They ascribed this abnormality to the lower reversibility of the stress-induced HCP martensite in the Fe–14Mn–5.5Si–8.5Cr–5Ni alloy due to its lower yield strength, which was estimated by the extrapolation of the  $\sigma_{0.2}$  at elevated temperatures. The lower yield strength means the easy occurrence of dislocations slip, which reduces the reversibility of the stress-induced HCP martensite. The results in Fig. 9 also showed that the yield strength of the deep-cooled Co<sub>30</sub>.30Ni alloy was higher than that of the Co<sub>2</sub>.19Al alloy. The abnormality between the SME and the content of stress-induced HCP martensite in the deep-cooled Co<sub>30</sub>.30Ni alloy and the Co<sub>2</sub>.19Al alloy can be ascribed to the difference between their yield strength.

## 5 Conclusions

1) The SME in both the CoAl and CoNi alloys showing FCC→HCP martensitic transformation results

from the stress-induced HCP martensite and its reverse transformation. The much higher SME in the solution-treated CoAl alloy than in the solution-treated CoNi alloy can be ascribed to its much higher yield strength due to solution strengthening of Al. The reason is that much higher yield strength can guarantee more stress-induced HCP martensite during deformation.

2) The strengthening effect of the thermal HCP martensite is very remarkable. The yield strength of the solution-treated Co<sub>30</sub>.30Ni alloy increases by 100 MPa after the intrusion of 10% thermal HCP martensite. This is the reason why the SME in the solution-treated Co<sub>30</sub>.30Ni alloy can be significantly improved after deep-cooling at 77 K.

3) The role of thermal HCP martensite in the CoAl shape memory alloys is also strengthening of matrix, as it does in the CoNi alloy. The decrease of the yield strength in the CoAlX alloys due to the drop in the content of thermal HCP martensite should be responsible for the deterioration in the SME with the drop in the content of thermal HCP martensite.

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## 热诱发马氏体对 CoAl 和 CoNi 合金形状记忆效应的作用

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**摘 要:** 为了明确热 HCP 马氏体对 CoAl 和 CoNi 合金形状记忆效应的作用, 研究 CoAl 和 CoNi 合金形状记忆效应(SME)与应力诱发马氏体和热 HCP 马氏体之间的关系。采用原位金相观察合金深冷前后热诱发马氏体的变化, 并研究其对应力诱发马氏体和形状记忆效应的影响。结果表明, CoAl 和 CoNi 合金的形状记忆效应都来源于应力诱发 HCP 马氏体, 热马氏体对两者形状记忆效应的贡献都是强化基体。CoAl 合金形状记忆效应高于 CoNi 合金的原因是 Al 原子对基体更强的固溶强化作用导致 CoAl 合金基体强度高于 CoNi 合金强度。

**关键词:** CoAl 合金; CoNi 合金; 形状记忆效应; 热 HCP 马氏体; 应力诱发 HCP 马氏体; 固溶强化

(Edited by Wei-ping CHEN)