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Mechanism and reversible behavior of the $\alpha' \rightarrow \gamma$ transformation in 1Cr18Ni9Ti stainless steel

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Focusing on the $\alpha' \rightarrow \gamma$ reverse transformation in pre-deformed 1Cr18Ni9Ti stainless steel, the present paper represents the different stages of the reverse process controlled respectively by nucleation-growth and shear transformation mechanisms. Thermal analyses combined with ferromagnetic measurements were used to investigate the whole process and to give their temperature ranges. It was found that the degree of deformation might take roles in the acting of the reverse mechanism, and the shear type transformation can benefit from the increase of reduction. For 90% prior deformation, the nucleation-growth reverse transformation began at about 583 K, while the shear transformation occurred between 763 and 883 K. The obtained structure underwent significantly recovery and recrystallization at 923 K. Part of the austenite obtained by shear-type reverse transformation would transform back again on cooling to room temperature in annealing. And an unusually reversible behavior during cyclic thermal process was detected. Ferromagnetic analysis and TEM observation showed that this reversible behavior could be damaged during the process of recovery and recrystallization.

1. Introduction

In the past two decades, the grain-refining technique through $\gamma \rightarrow \alpha' \rightarrow \gamma$ transformation has been developed to strengthen the popular commercial Fe-Cr-Ni austenitic steels [1–5], where the reverse transformation of deformation-induced martensite ($\alpha' \rightarrow \gamma$) was considered to play a decisive role in formation of the final austenic structure. But there has been no distinct idea shaped about the mechanism of $\alpha' \rightarrow \gamma$ reverse transformation from deformation-induced martensite so far. Tomimura *et al.* suggested that the reverse process depended greatly on the composition of steels and the annealing temperature, and for 18–9 austenitic steels, α' reverted to γ by a diffusion nucleation-growth process when annealed at 923 K [6, 7]. This diffusion mechanism was also identified by Katoh *et al.* on the study of AISI304 stainless steel [8]. While in the study of Jamekata *et al.*, a shear reverse transformation was reported to occur in the temperature range from 583 to 883 K in AISI304 steel [9]. Thus a system study of the reversion will be beneficial. In the present work, dynamic measurements were used aiming to disclose the detailed process of reversion in the same system of alloys. The result shows both mechanisms of reversion can compete to take roles, and part of austenite produced by shear reversion will transform back to secondary martensite and exhibit a reversible behavior. The reason for the formation of this kind of secondary martensite was discussed in this paper.

2. Experimental

The material used in this study is commercial 1Cr18Ni9Ti sheet steel with composition (wt%): 0.12C, 18.17Cr, 10.14Ni, 0.53Ti and balance Fe. The sheet was first solution-treated at 1473 K for 30 min and then chemically cleansed to remove the oxidized surface layer. After rolled at room temperature in a four-roll mill under water-jet cooling with each rolling pass limited to ensure a uniform deformation in thickness, the sheet with full martensitic structure (>90% α') was cut to proper sizes for various tests. Measurements of the reverse transformation temperatures were taken on Du Pont 2000 DSC (Differential Scanning

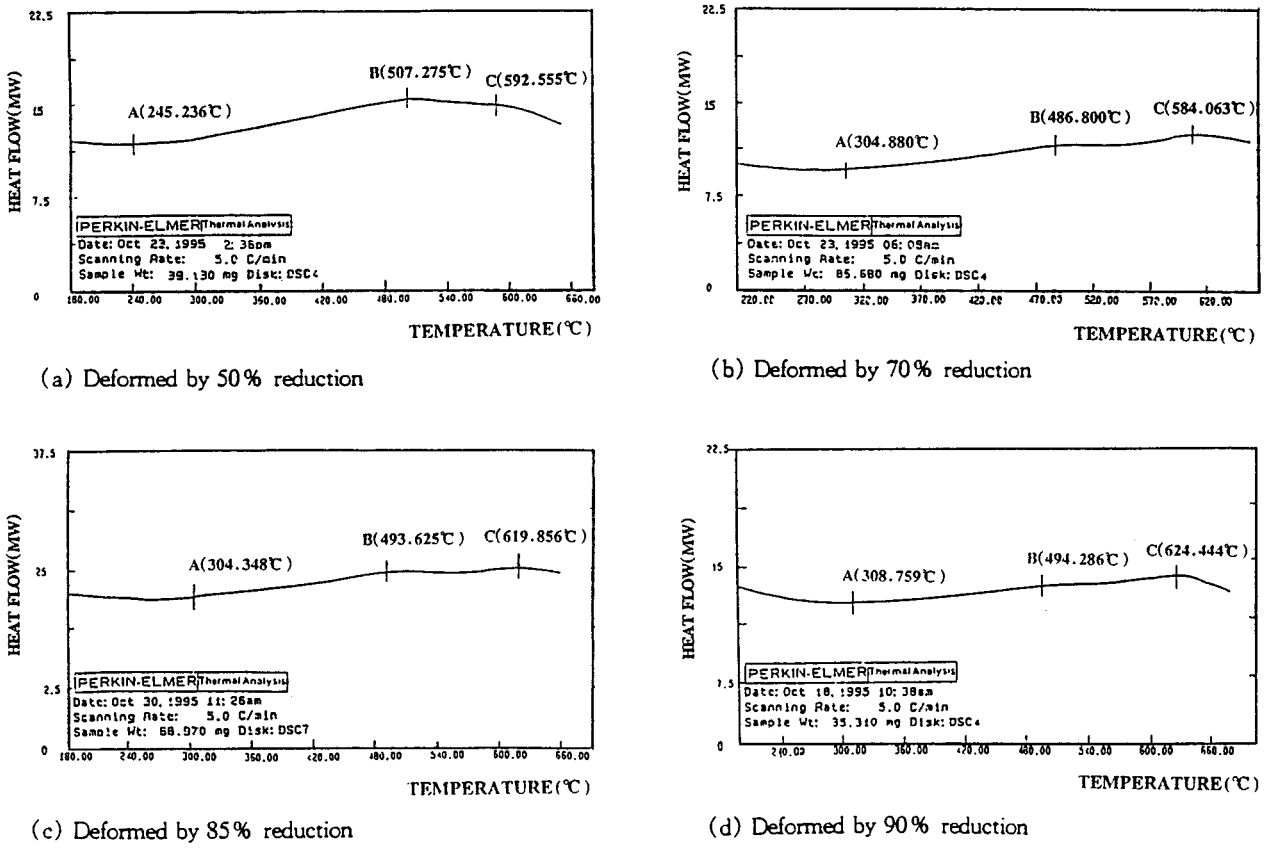


Figure 1 The DSC curves to show the continuous heating course for samples of different reductions.

Calorimeter) analyzer together with LDJ Model 9600 VSM (Vibrating Sample Magnetometer). The scanning was controlled at a rate of 5 K/min for the DSC tests, and for the VSM measurements the minimum possible heating and cooling rate provided by the instrument were 20 K/min. For TEM (Transmission Electron Microscope) and XRD (X-ray Diffraction) measurements, samples were further annealed in salt baths at various temperatures for various periods of time and then cooled in air. The reverse-transformed structures obtained from annealing were observed by JEM-200CX transmission electron microscope operating at 200 kV. And the γ contents were determined with the standard X-ray diffraction technique carried out on D/max-3A diffractometer using CoK_α radiation with the operating parameters being 40 kV, 25 mA, 0.05 °C/s.

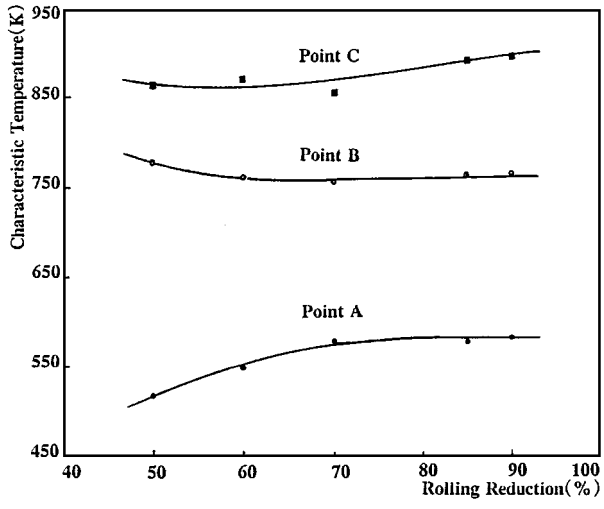


Figure 2 Variation of characteristic temperatures shown in Fig. 1 as a function of rolling reduction.

3. Results

3.1. Determination of the reverse transformation temperature range

Fig. 1 shows the results of DSC analyses for samples of different reductions. Although the slope of the curves of these materials developed gradually over a broad temperature range, two peaks with three characteristic points were identified for each curve. The characteristic points (points A, B, C) have their tendency to approach a fixed value with the increase of reduction, which is summarized in Fig. 2.

Results from dynamic VSM measurement may help to explain the characteristic points. Fig. 3 shows the plot of magnetic property vs. temperature through the continuous heating and subsequent cooling process when a sample containing 90% α' phase was annealing from 300 to 900 K. From this figure, it can be found that the sample lost its magnetism completely when the temperature reached above 883 K, indicating the end of the $\alpha' \rightarrow \gamma$ transformation. The result agrees with the values reported in previous work [9]. Thus the presence of the second peak in Fig. 1 is likely to be associated with a process other than $\alpha' \rightarrow \gamma$ reverse transformation. TEM observation revealed that a second precipitation just occurred at the temperature range, so the curve of point C in Fig. 2 stands for the temperature of significant precipitation. Also in Fig. 3, if regardless of the measure errors, the temperature where the rapidest change of magnetism occurred (computed to be 755 K)

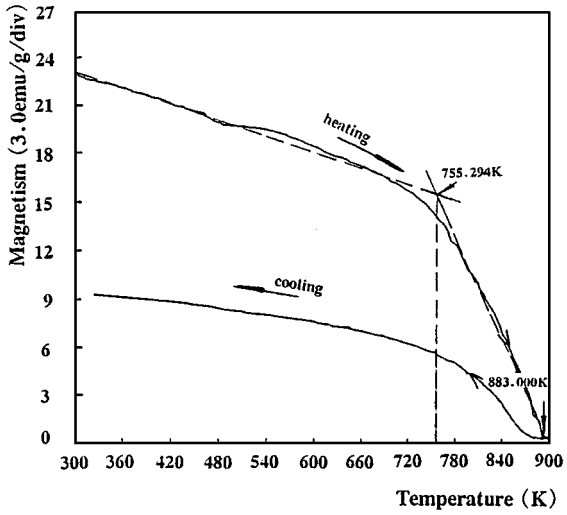


Figure 3 The change of magnetic property of the sample rolled by 90% reduction as a function of temperature during annealing.

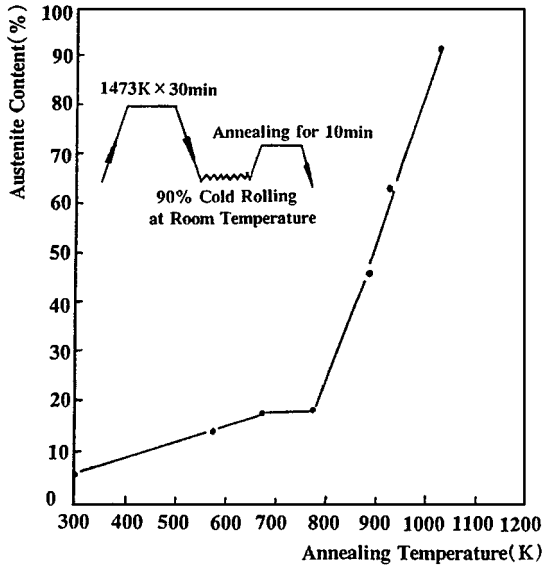


Figure 4 The amount of reverse-transformed austenite determined by X-ray analyses as a function of annealing temperature.

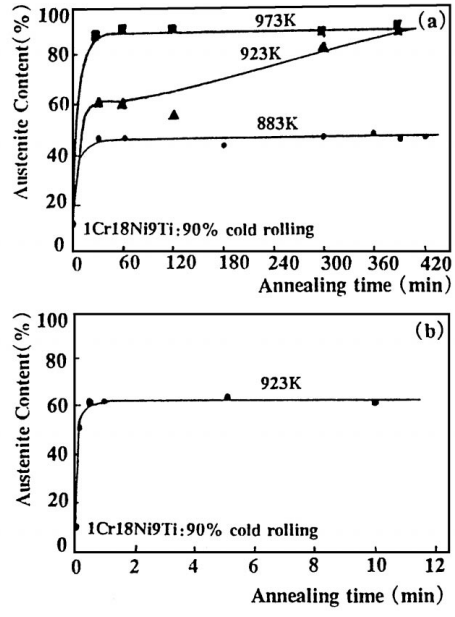
was coincides with the stable value of point B on DSC curve (approaching 763 K), suggesting that the point B on DSC curve is the temperature of accelerating the reverse transformation.

So for 90% cold rolling specimens, during a continuous heating process, the reverse transformation of α'-martensite began at about 583 K (the stable temperature of point A as shown in Fig. 1), went through a gradual transformation stage, then entered into an accelerating stage at about 763 K, and finally finished at 883 K. Fig. 4 is the plot of austenite content as a function of temperature, measured by XRD analysis on specimens cold-rolled by 90% reduction and stepwise annealed for 10 min at a series of temperature from 300 to 1100 K, showing obviously a bent line consisting of a gradual stage and a steep stage of the austenite formation. Therefore we can deduce that the two formation stages of austenite presented above reflect the kinetic characteristics of the transformation by different mechanisms. That is, the $\alpha' \rightarrow \gamma$ transformation is progressing successively through a nucleation-growth transformation (also defined as diffusion transformation by other papers) and a shear transformation during an uninterrupted heating process. Since 583 K, the deformation-induced martensite transforms to austenite slowly by nucleation-growth transformation because the process of nucleation and growth is time-dependent, so the amount of γ structure obtained is small after a short time annealing. But when the temperature reaches 763 K, shear mechanism will act and result in a sudden acceleration of austenite formation till 883 K. Therefore the experimentally determined temperatures for the starting point of nucleation-growth reverse transformation (A_s point), and the starting and finishing point of shear reverse transformation (A'_s, A'_f point) are 583, 763, 883 K, respectively. In this investigation it is difficult to identify the finishing point of the nucleation-growth reverse transformation (A_f point) due to the superposition of two stages by different reverse mechanisms. The values of A'_s, A'_f point agree with the result obtained by Jamekata *et al.* [9].

3.2. Mechanism of reverse transformation on condition of refining annealing

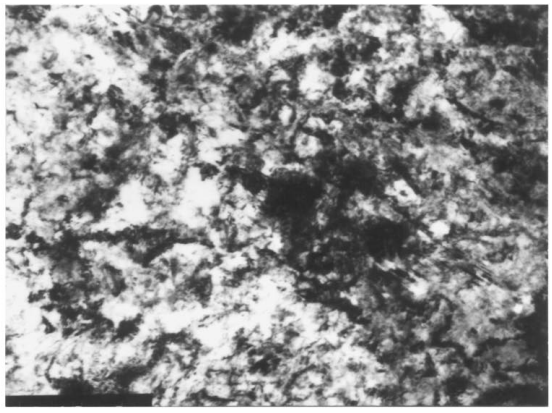
In general, annealing for transformation refinement is carried out just above the finish temperature of $\alpha' \rightarrow \gamma$ transformation in order to achieve a full ultra-fine austenite structure. In our case, the annealing temperature was set above 883 K, which made the occurrence of shear transformation possible.

Fig. 5 shows the kinetic curves of the austenite formation vs. holding time when specimens with 90% cold worked structure were annealed above A'_f point. The feature that the shear formation of austenite is time-independent is clearly revealed here except for annealing at 923 K for a long times. For 923 K annealing, Fig. 5b shows the beginning part of its kinetic curve on

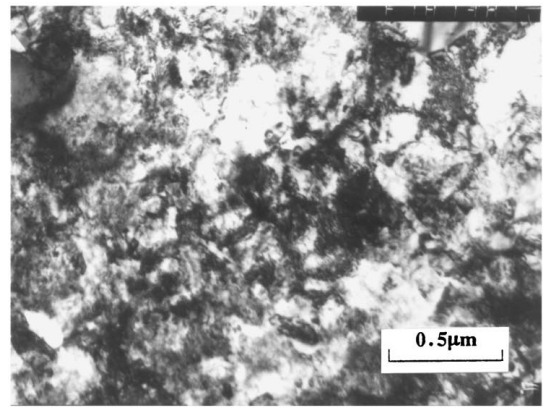


* The specimen annealed at 923K for 120min is observed to have a heavy precipitation of second phase during annealing.

Figure 5 The amount of reverse-transformed austenite determined by X-ray analyses as a function of holding time. (a) holding at 883, 923, 973 K for long annealing; (b) holding at 923 K for short annealing.



(a) The parent structure with 90% deformation induced martensite before annealing.



(b) The reverse-transformed structure after refining annealing, annealed at 883K(A_f' point) for 15hrs

Figure 6 Comparison of TEM micrography between two structures produced by $\gamma \rightarrow \alpha'$ process and its reverse process respectively.

another scale, demonstrating that the time-independent feature still keeps well for short annealing. Explanation to this unusual case at the temperature will be given from the finding discussed later.

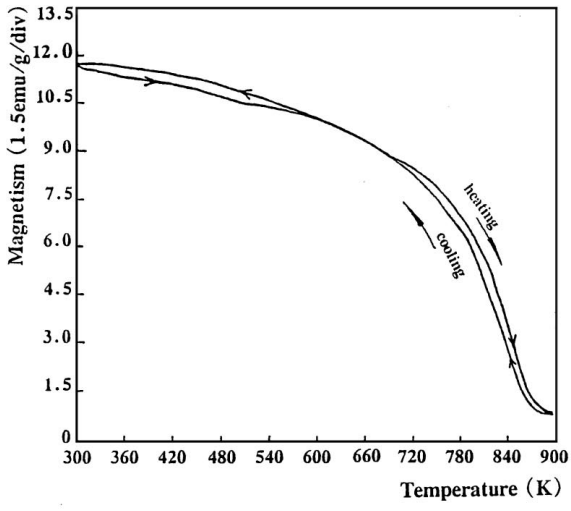
The shear mechanism of reverse transformation is further corroborated by TEM observation as shown in Fig. 6. It can be seen that the reverse-transformed structure obtained by annealing at A_f' point, even if the holding time reached 15 h, still contained a high density of dislocations and revealed a structure without well-defined grain boundaries, the feature of which is the same as that of the microstructure obtained by shear reversion in previous studies [7, 9]. But it should be noted that at this temperature, both types of transformation mechanism have their possibility of operation from the view of thermodynamics, and the shear mechanism will be dominating due to its superiority in kinetics. But due to the refining mixed structure, it is difficult to make sure the different microstructure produced by two mechanisms respectively. And it is also hard to distinguish the morphology of structure reversed by diffusion mechanism from the structure after a full-recrystallization.

3.3. The reversible behavior of $\alpha' \rightarrow \gamma$ reverse transformation

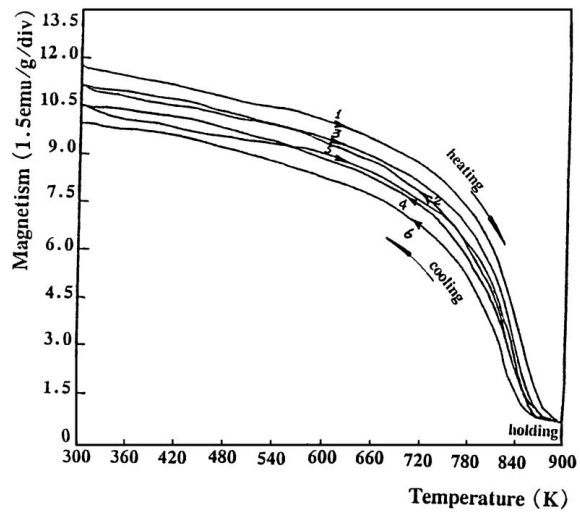
From the above result, we took 883 K as the finish point of the shear $\alpha' \rightarrow \gamma$ reverse transformation according to the magnetic property (Fig. 3). But we find that the austenite content was much lower than 90% even after annealing at 883 K (A_f' point) for 15 h. With reference to the cooling curve shown in Fig. 3, we deduce that a part of the austenite may transform back again during the subsequent cooling, and may result in the increase of the part of ferromagnetic martensite.

In order to verify the existence of the unstable reverse-transformed austenite, we monitored the variation of magnetism vs. temperature through the whole thermal cycles including heating, holding, cooling. Samples used here had been annealed at A_f' point for 6.5 h to undergo a complete $\alpha' \rightarrow \gamma$ reverse transformation.

Fig. 7a shows such a variation curve obtained after a complete thermal cycle without holding. As expected, the magnetic property of the samples changed with temperature, demonstrating the existence of the secondary transformation of martensite formed from the reversed



(a) One thermal cycle without intermediate holding stage



(b) Three continuous thermal cycles with each one added with 30 min holding stage

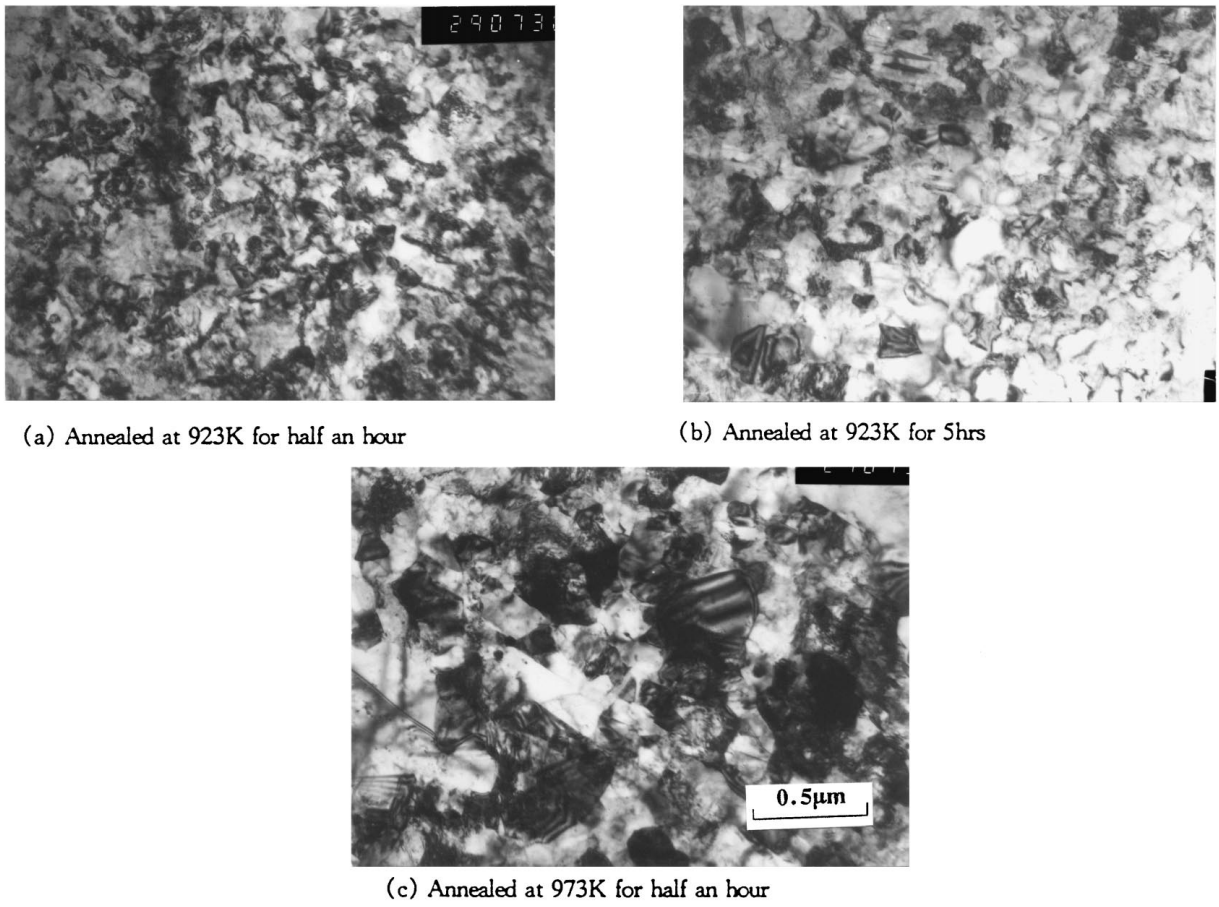
Figure 7 The variation of magnetism vs temperature through thermal cycles for samples pretreated with a complete reverse transformation.

austenite on cooling to room temperature. Particularly, the curve presented a well closed cycle and revealed a reversible feature of the $\alpha' \rightarrow \gamma$ transformation that appears in Fe-Cr-Ni alloy system. Further study shows that this reversible behavior can be damaged by holding at temperature above A'_f point, which is found in Fig. 7b, showing that the curve of three continuous thermal cycles with 30 min holding on each cycle cannot form itself into a closed loop. But up to now, it is difficult to distinguish the secondary martensite from the mixed grains without apparent boundaries.

4. Discussion

Generally, the formation of secondary α'-martensite is considered due to a higher M_s point near room temperature. However, according to the formulation by Eichelman and Hall [10], the M_s point of the steel is far below room temperature. Previous study on Fe-18Cr-7Ni-0.18C stainless steel also showed the experimental M_s, M_d points were 190, 360 K, respectively [11]. So a possible explanation is that the changes in composition of γ phase caused by precipitation of carbides or secondary phases may result in the increase of M_s point to room temperature [12]. However, no consistent relationship was observed between the occurrence of the secondary martensite transformation and the precipitation of secondary phases. In fact, at 883 K that the special behavior was found there was no precipitation being detected by X-ray analyses, while in the temperature of 923 K at which severe precipitation occurred observed by TEM, the reversible behavior of secondary martensite transformation was notably broken-down. The situation of precipitation is also reflected from the curve of point C in Fig. 2. In that case, the composition of γ phase is not considered the factor of controlling the stability of austenite.

Based on the experimental results, we deduce that the reversible behavior of $\alpha' \rightarrow \gamma$ transformation is to great extent related to the crystallographic structure of product austenite which possibly keeps good coherent interfaces with its parent martensite due to the shear mechanism. So from the point of the thermodynamics it will be not difficult for a secondary martensite transformation to occur. From Fig. 2, we can observe that the starting point of shear reversion (point B) declines and that of diffusion reversion (point A) rises with the increase of the deformation. That is to say, the degree of deformation seems to take roles in the acting of the reverse mechanism. The shear type of reverse transformation will be dominating with the increase of deformation. Furthermore, there is a decrease tendency of thermal energy released by transformation with the increase of reduction, i.e. the increase of the dislocation density, as shown in Fig. 1. The lower transformation heat is thought of the typical features in the reversible transformation such as $\gamma \rightarrow \varepsilon$ elastically martensitic transformation [13]. Therefore a high density of dislocations may play an important role in the occurrence of reversible $\alpha' \rightarrow \gamma$ transformation. This kind of reversible behavior should be kept only if the structure of dislocation and the coherent relation between parent



(a) Annealed at 923K for half an hour

(b) Annealed at 923K for 5hrs

(c) Annealed at 973K for half an hour

Figure 8 TEM micrographs showing the microstructure changes of reverse-transformed specimens with different annealing procedures.

and product phases are destroyed by a thermal course such as recovery and recrystallization and thereby the reverse-transformed austenite will be effectively stabilized.

XRD analysis, usually performed at room temperature, although can not give the real content of the reverse-transformed austenite and led to the seeming abnormal phenomenon as shown in Fig. 5a, provided a fact that the austenite content at room temperature was gradually recovering to higher levels by holding at 923 K for longer time periods. Finally a nearly full-austenite structure (90% γ) was obtained when the sample was annealed at this temperature for 5 h or more. The higher the annealing temperature, the shorter the holding time necessary for achieving a complete austenitic structure (90% γ) at room temperature. Samples annealed at 973 K were seen to develop a complete austenitic structure in less than half an hour.

Further support comes from the observation of the corresponding microstructures. TEM analyses show that the regaining of austenite content was well consistent with the recrystallization process of the reverse-transformed structure as shown in Fig. 8, which revealed that a visible recrystallization happened to begin at 923 K, and was almost completed after 5 h annealing. When the annealing temperature increased to 973 K, the holding time for completing the recrystallization can be greatly shortened and half an hour was enough to form perfect full-equiaxed grains. Therefore, samples annealed at 973 K all essentially had full-austenitic structure and no unstable austenite remained.

5. Conclusion

During a continuous heating process, the $\alpha' \rightarrow \gamma$ reverse transformation in 1Cr18Ni9Ti stainless steel are found to be carried out in two successive stages controlled by different mechanisms of reversion. The degree of deformation may take roles in the acting of the reverse mechanism. And the shear type transformation will benefit from the increase of reduction, which can easily occur with the increase of dislocation density. For 90% deformation specimens, the nucleation-growth transformation began from 583 K, and the shear reverse transformation occurred in the temperature range from about 763 to 883 K. Hence, the reverse process of the grain-refinement for 1Cr18Ni9Ti steel was finally carried out by shear mechanism, and resulted in parts of secondary martensitic transformation on cooling to room temperature. So a false impression of the reverse transformation kinetics of $\alpha' \rightarrow \gamma$ at 883 K (Fig. 5a) was created if measured by XRD in static manner. The secondary martensitic transformation is different from the usual one caused by the change of austenitic composition by exhibiting a reversible feature. The reversible behavior is considered to relate with the increasing of dislocation. And less energy as shown in the reversion from structure with high density of dislocation may contribute to the special behavior of the $\alpha' \rightarrow \gamma$ transformation in the Fe-Cr-Ni alloys. Present results show that the process of recovery and recrystallization will damage it. Therefore, at room temperature the amount of austenite in the annealed specimens will rise again to normal level when recrystallization process is thoroughly conducted as observed in the case of 923 K annealing.

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