

The effects of cold work on the ordering reaction in Alloy 600

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1. Introduction

Recently it is reported that there is ordering reaction in Alloy 600 and that the exothermic reaction near 500°C in the water quenching specimen originated from formation of short range order (SRO) [1].

Marucco and Nath [2] have investigated and concluded that most commercial Ni–Cr based alloys like Alloy 600 have an ordering reaction based on Ni₂Cr at temperatures below 550°C. Ordering is known to have the following consequences: (i) dimensional instability due to lattice parameter changes; (ii) electrical and thermal characteristics; (iii) changes in mechanical properties due to pile-ups of dislocations; and (iv) phenomenon of negative creep or material contraction under load.

Completing the picture further, Lang et al. [3] found that order and precipitation phenomena in Ni–Cr based alloys is strongly dependent on thermo-mechanical treatments producing different metallurgical states. Slow cooling from solution annealing temperatures produces a rather advanced precipitation stage and a degree of SRO corresponding to the thermodynamic equilibrium at 500°C, while a more disordered state is retained.

The kinetics of SRO formation is strongly determined by the nucleation of ordered zones, and carbon in the solution appears to suppress the formation of SRO nuclei by trapping excess vacancies during water quenching and hence, retarding the establishment of SRO. Cold working destroys SRO but also produces various lattice defects, which enhance SRO formation and carbide precipitation during re-heating.

Marucco [4] has established the existence of an order–disorder transformation in Ni–Cr base alloys from the study of Ni₂Cr, Ni₃Cr, and Ni–Cr–Fe. She concluded that the degree of order primarily depends on Cr content and that the ordering kinetics become slower when the Ni:Cr atomic ratio departs from that of stoichiometric Ni₂Cr. The presence of Fe has a strong delaying effect on ordering kinetics, even in small quantities and other alloying elements can also influence the transformation.

It has been ascertained that the limited lattice contraction accompanying SRO markedly increases owing to long range order (LRO), while the resistivity decreases sharply and the LRO kinetics are markedly delayed by Fe addition [5]. The equilibrium degree of LRO depends on temperature and Cr concentration.

The SRO–LRO transition can be confirmed by the appearance of appreciable variations in the physical property like lattice contraction.

In this study, the specific heat variation with temperature, amount of energy released due to the exothermic reaction, and activation energy for the exothermic reaction were systematically investigated to understand how the cold work affect ordering reaction in Alloy 600.

2. Experimental

The Alloy 600 used in this study was a mill-annealed rod with a diameter of 10 mm. The specimens used for the DSC measurements were water quenched after heat treatments at 1095°C for 1h.

Some specimens were water quenched from 1095°C and, then, were water quenched and furnace cooled after thermal treatment at 700°C for 32 h. These were then cold worked up to 30%. The latter treatment was chosen to precipitate all carbon as carbide. In addition, some specimens quenched from 1095°C were aged at 474°C for 1100 and 10000 h, subsequently.

Thermal analyses were preferred on specimens about 5 mm in diameter and 2 mm in thickness and carried out from room temperature up to 800°C. The scan rate of the thermal analyses was generally 5°C/min.

3. Results and Discussion

In Fig. 1, the typical curves of the thermal analyses show the temperature ranges of the exothermic and the endothermic reactions. These reactions were observed at about 430–520 and 520–590°C, respectively, for a specimen quenched after a 1095°C–1/2h treatment at a heating rate of 5°C/min. However, for the same specimen after a DSC measurement (this may be the same as a furnace-cooled specimen since this specimen is cooled in furnace), only an endothermic reaction was observed.

The exothermic reaction was observed at 451–521°C and at 440–523°C in specimens quenched after 647°C–1h and 700°C–1h treatment, respectively, although these curves are not shown because those curves are almost same as Fig. 1 except temperature ranges. It is reported that carbides are precipitated along the grain boundaries after 647°C–1h and 700°C–1h treatment [6].

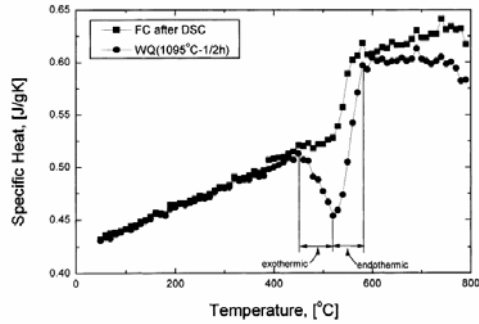


Fig. 1. Specific heat variations with temperature for Alloy 600 specimen water quenched after 1095°C-1/2 h treatment (solid circle) and furnace cooled after DSC (solid square).

The thermal analysis results on a 30% cold worked specimen after a water-quenching from a 700°C-32h treatment, and on the same specimen furnace cooled after DSC measurements are shown in Fig. 2. Here, the exothermic and the endothermic reactions in cold worked specimen were at about 60-390 and 480-530°C, respectively, and the endothermic reaction behavior in furnace cooling specimen is very similar to that for the furnace cooled specimen after a 1095°C-1/2h treatment.

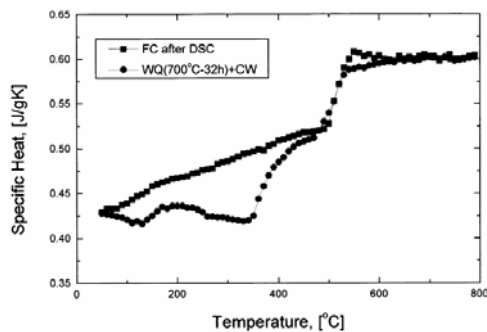


Fig. 2. Specific heat variations with temperature for the 30% cold worked specimen after water quenching from 700°C-32-h treatment (solid circle) and furnace cooled specimen after DSC (solid square).

On the other hand, as shown in Fig. 3, the result for the 30% cold worked specimen after furnace-cooled from 700°C-32h showed that the exothermic and endothermic reactions appeared at 180-390 and 430-530°C, respectively.

The amounts of energy released from the quenched specimens increase roughly with the heat-treatment temperature as follows: 6.7 J g⁻¹ from the specimen treated at 647°C-1 h; 6.7 J g⁻¹ at 700°C-1 h; 10 J g⁻¹ at 764°C-1 h; 7.9 J g⁻¹ at 811°C-1 h; 8.4 J g⁻¹ at 874°C-1 h; and 10 J g⁻¹ at 1095°C-1/2 h.

Furthermore, the temperatures of both reactions were slightly dependent upon the thermo-mechanical history of the specimens and heating rates.

It is clear that the cold work lowers the ordering temperature near 100°C and enhances ordering reaction in Alloy 600. This means that increase in number of dislocations by slip due to the cold work provides a

proper environment for the ordering reaction at lower temperature.

The exothermic reaction in Fig. 2 and 3 is understood to be annihilation of dislocation and formation of the SRO. There are two peaks near 130 and 330°C. Although it is not possible which peak is due to formation of SRO at the present, both annihilation of dislocation and formation of SRO are taking place below 330°C.

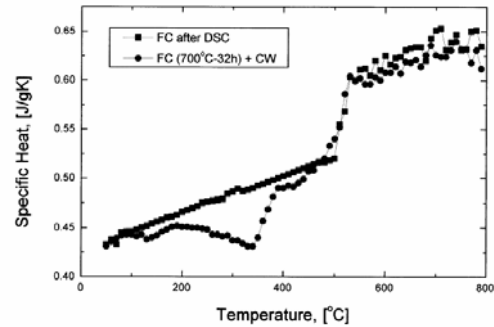


Fig. 3. Specific heat variations with temperature for the 30% cold worked specimen after furnace cooled from 700°C-32-h treatment (solid circle) and furnace cooled specimen after DSC (solid square).

4. Conclusion

The exothermic and endothermic reactions in Alloy 600 observed during thermal analyses are due to the ordering and disordering reactions, respectively. The temperatures for the ordering reactions were observed to vary with the thermo-mechanical treatment of the alloy, and the ordering reaction temperature can be significantly lowered by cold work. It is confirmed that the ordering reaction occurred below 100°C for the cold worked material regardless of the prior treatment conditions. It seems that the cold work provides an additional energy for the ordering reaction at relatively lower temperature.

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