EVALUATION OF THE AUSTENITIC GRAIN GROWTH BY THERMOELECTRIC POWER MEASUREMENTS

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Abstract. The influence of the deformation grade on the recrystallised grain size has been studied in the AISI 304 stainless steel. Therefore, cold rolled samples of this material with reductions varying between 30% and 80% were annealed at different temperatures and subsequently quenched. The mean austenitic grain sizes were measured and compared. Moreover, the correlation between the variation of the thermoelectric power and the grain growth was investigated for each degree of prior deformation.

Introduction

Austenitic stainless steels AISI 304 are widely used steels combining high corrosion-oxidation resistance, good heat resisting properties and a relative low cost. However, one of the main drawbacks of these steels is their low mechanical resistance obtained in standard production processes. Therefore, in recent years an increasing interest can be noted in the development of new processing routes. One of the key issues in this field is the study of the microstructural evolution of the cold-rolling material at different annealing temperatures.

In this context, the aim of the work presented in this paper was to characterise the recrystallised grain growth of the AISI 304 steels annealed at temperatures between 1000 and 1200 °C and for different rolling reductions, using thermoelectric power (TEP) measurements.

Experimental Procedure

The chemical composition of the studied steel in wt% is as follows: 0.039% C, 0.22% Si, 0.001% S, 0.029% P, 1.15% Mn, 18.0% Cr, 8.52% Ni, 0.24% Mo, 0.25% Cu, 0.0005% B, and 0.04% N. The material was obtained from a hot-rolled coil and was subsequently cold rolled down to 30%, 50%, 70% and 80% reduction. In order to study the evolution of the recrystallised grain growth, samples 30 mm in length, 2 mm in width and 0.8 mm in thickness were annealed at different temperatures (1000, 1050, 1100, 1150 and 1200 °C). After 180 seconds of holding time, specimens were rapidly cooled to room temperature. These heat treatments were appropriately chosen in order to obtain fully recrystallised samples in each case. The samples were etched with a solution containing 60% hydrochloric acid, 20% nitric acid and 20% methanol. Fig. 1 shows the optical micrographs of the annealed microstructures are shown for the case of 50% deformation. The micrographs were then analysed with the Image Tool 2.0 software.

Fig. 1. Optical micrographs of the AISI 304 steel with 50% rolling reduction (a) in the as-cold rolled condition and after heat treatment at (b) 1000 °C, (c) 1050 °C, (d) 1100 °C, (e) 1150 °C and (f) 1200 °C.

A schematic representation of the TEP apparatus is given elsewhere [1]. The experimental procedure of the TEP measurement is the following: the sample is pressed between two blocks of a reference metal (in this case, pure copper). One of the blocks is at 15 °C, while the other is at 25 °C to obtain a temperature difference ΔT . A potential difference ΔV is generated at the reference metal contacts. The apparatus does not give the absolute the TEP value of the sample (S^*), but a relative TEP (S) in comparison to the TEP of pure copper (S_0^*) at 20 °C. S is given by the following relation:

$$S = S^* - S_0^* = \frac{\Delta V}{\Delta T}.$$

The TEP value does not depend on the shape of the sample, which is a great advantage of this technique. Moreover, the measurement is performed very quickly (less than 1 min) and precisely (about $\pm 0.5\%$). The resolution is of the order of $0.001 \mu\text{V/K}$.

Results and Discussion

The evolution of the microstructure in each annealing cycle has been evaluated using relative TEP measurements. Thus, for each degree of deformation the TEP value corresponding to the lowest annealing temperature tested (1000°C) was chosen as the reference level for the study of the recrystallised grain size evolution. Fig. 2a shows the evolution of the recrystallised grain size (expressed as the average grain diameter) with the annealing temperature, whereas in Fig. 2b the effect of the annealing temperature on the TEP measurement is represented.

Fig. 2a. Evolution of the recrystallised grain size for the four cold rolling reductions

It can be seen that the evolution of the grain size in the 30% and 50% deformed material is quite similar, albeit the TEP values at 50% reduction are slightly higher at lower annealing temperatures compared to the TEP measurements at 30% deformation. The negative values of the TEP at lower holding temperatures and smaller degree of deformation indicate that in these cases the TEP measurement is dominated by other effects than recrystallised grain growth, e.g. dissolution processes. At higher temperature a clear influence of the grain growth can be noted, especially in the highly deformed steels.

The faster kinetics of the higher deformed material (70 and 80%) leads to higher recrystallised grain sizes and higher values of TEP compared to the case of small deformations. However, at 1200°C a higher grain size of the 70% deformed material is measured compared to the 80% deformed steel, which is also reflected by a higher TEP value at that temperature. This behaviour could be related to

dissolution processes of pinning particles such as $M_{23}C_6$ during annealing in the 70% deformed steel [2].

Fig. 2b. Evolution of the thermoelectric power for the four cold rolling reductions

Another interpretation could be that the fast nucleation in the 80% deformed material favours rapid impingement, leading to a final grain size that is smaller than in the case of 70% reduction [3].

Conclusions

A number of isothermal annealing treatments at 1000, 1050, 1100, 1150 and 1200 °C were carried out in AISI 304 stainless steels with reductions between 30 and 80%, with the aim of studying the evolution of the recrystallised grain growth. At lower annealing temperatures the increase of the grain size is still relatively low and only a minor influence on TEP is observed, since the contribution of grain size to TEP is overshadowed by other effects, such as the dissolution of particles. At higher annealing temperatures an optimum balance is found in the 70% deformed steel between a fast recrystallisation kinetics, a slow impingement rate and a weak particle pinning effect, which leads to the highest recrystallised grain size. This is also confirmed by the results of the TEP measurements.

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References

- [1] A. Ney Jose Luigi, *Metall. Mater. Trans. A* **29** (1998), p.2669.
- [2] T. Sourmail, *Mater. Sci. Technol.* **17** (2001), p.1.
- [3] F.J. Humphreys and M. Hatherly Green, *Recrystallization and Related Annealing Phenomena*, Pergamon Press, Oxford, UK (1995), p.205.