ISSN 1392–1320 MATERIALS SCIENCE (MEDŽIAGOTYRA). Vol. 13, No. 2. 2007

Effect of High-Energy-Density Pulse Current on Solidification Microstructure of FeCrNi Alloy

Fucheng ZHANG*, Ming ZHANG, Bo LV, Jianhui LI

State Key Laboratory of Metastable Materials Science and Technology, Yanshan University, Qinhuangdao 066004, P. R. China

Received 13 August 2006; accepted 21 March 2007

High energy density pulse current (HEDPC) treatment was used to refine the solidification microstructure of the austenite-ferrite two phases Fe70Cr18Ni12 alloy. It show that the austenitic grain size of the alloy can be reduced in two orders of magnitude to 0.6 μm from 120 μm, and the size of the ferrite phase can be refined to nanometer scale after the HEDPC treatment; the suitable start temperature of the HEDPC treatment is at about melting point of the alloy, the HEDPC treatment can change the relative content of austenite and ferrite phases of the alloy.
Keywords: pulse current, microstructure, solidification, FeCrNi alloy, refinement.

INTRODUCTION

The electromagnetic field has been widely used in industry because of its high efficiency, economic benefits and cleanliness. The current can affect the following aspects of the solidification microstructure of metal: refining solidification microstructure, eliminating macro segregation, increasing content of alloying elements within grains, decreasing residual stress, avoiding cracks and improving surface quality [1 – 3].

An Indian scholar named Mistra published the earliest report about the effect of an electromagnetic field on solidification microstructure of metal in 1986. He let 40 MA/cm^2 direct current pass through liquid $Pb_{68}Sb_{15}Sn_7$ eutectic alloy and $Pb_{87}Sb_{10}Sn_3$ sub-eutectic alloy and found that the microstructure of the alloys was fined after solidification [4]. Afterwards, Nakada. let pulse current pass through $Sn_{85}Pb_{15}$ alloy and found that beyond the refinement of the solidification microstructure, there was a change from dendritic crystal to spherical crystal [5]. Barnak. tested the influence of the HEDPC on the solidification microstructure of $Pb_{60}Sb_{40}$ and $Pb_{63}Sb_{37}$ alloys. His result proved that pulse current could increase overcooling degree of metal, and reduce the eutectic grain size, moreover, the grain size decreased with the increase of pulse current density [6]. Later, the same conclusion was obtained by applying this technique to A1 alloys[3, 7].

From the view of the results on this research area [3 – 9], most materials applied for the study were metal with low melting point, also the employed current density was low, thus, the test is easier. For high melting point metal or alloy, such as iron and steel materials, and for HEDPC the test is more difficult. And also it has more requirements for experimental equipment, all of these increases the difficulty of study. The present investigation aimed at studying the influence of pulse current on the solidification microstructure of Fe70Cr18Ni12 alloy.

EXPERIMENTAL

A HEDPC equipment and a crucible furnace with a bottom door were designed and made. The HEDPC equipment consisted of three shunt capacitances with 100 μF as energy-saving device and a inductance coil used to adjust the frequency and density of the output pulse current. Its maximum charging voltage, the maximum pulse current and the pulse current frequency were 40 kV, 100 kA and within 5×10^3 Hz – 100×10^3 Hz, respectively. The crucible furnace with a bottom door is shown in Fig. 1. The reason of using the bottom door was to raise the cooling rate of the sample prepared and to avoid the microstructure change of the solidified alloy caused by slow cooling in the furnace. A pure molybdenum stick with high-melting point and good electric conductivity was chosen as electrode material. Argon was used as protection gas. A TDS-3012 digital phosphorescence oscilloscope was used to test the parameters of the pulse current in the process of discharge. Neophot-21 optical microscope, H-800 TEM and D/max-rB X-ray diffractometer were used to analyze the solidification microstructure of the alloy.

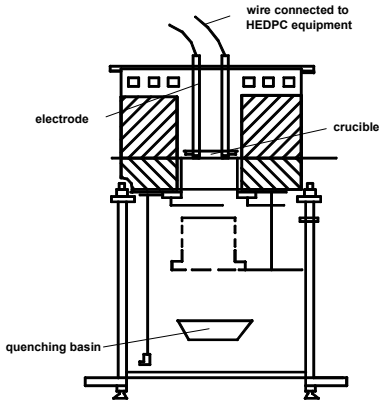

Fig. 1. Schematic drawing of crucible furnace

A FeCrNi alloy was used in this study, whose composition (wt-%) is Cr 18.06 %, Ni 12.10 %, Fe 69.77 %, which approaches the eutectic composition of FeCrNi ternary alloy [10]. Lots of studies indicated that it was easy to obtain ultrafine crystallization microstructure for eutectic composition alloy. Umeda has approved this standpoint by exploring the effect of different cooling condition on the solidification microstructure of eutectic composition alloy [11]. According to the DSC curve test,

* Corresponding author. Tel.: +86-335-8063949; fax.: +86-335-8051899.
E-mail address: *zfc@ysu.edu.cn* (F. Zhang)

the beginning and the finishing solidification temperatures of the alloy were at 1467 °C and 1441 °C, respectively.

Firstly, when the crucible furnace temperature was up to 1400 °C, the bottom door of the furnace was opened and a alumina crucible with the alloy tested put into the furnace. Afterwards, when the furnace temperature reached 1550 °C, the Mo electrode connected with the HEDPC equipment was put into the liquid alloy. After the furnace temperature lowered to 1470 °C, 1490 °C and 1520 °C, the HEDPC pass through the liquid alloy and stopped at 1400 °C. In the mean time, the bottom door of the furnace was opened to make the sample with alumina crucible and Mo electrode drop into water together for quick cooling. Because the discharging voltage in this study was very high, high-temperature gas was easily punctured, therefore a alumina tube was used to isolate the Mo electrodes which were in high-temperature gas area of the furnace during the experiment, so as to ensure HEDPC to pass through the liquid alloy and avoid the loss of the energy caused by gas spark through.

In order to investigate the effect of the HEDPC on the solidification microstructure of the alloy, different pulse current parameters had been taken into this study. The experimental conditions are shown in Table 1. The size of the specimen prepared in this study was $\varnothing 10\ \text{mm} \times 70\ \text{mm}$, the specimen was sectioned along vertical, and the section surface was polished and etched using the aqua regia to get the solidification microstructure. The TEM specimen was prepared following the standard grinding and ion-million procedures.

Table 1. Pulse current parameters tested

No.	Charging voltage (V)	Discharging frequency (Hz)	Peak current (A)	Current density (A/cm^2)
1	10×10^3	1×10^4	9×10^3	1.1×10^4
2	20×10^3	1×10^4	24×10^3	3.1×10^4
3	30×10^3	1×10^4	41×10^3	5.2×10^4

RESULTS AND DISCUSSION

Fig. 2 shows the solidification microstructures of the alloy before and after the HEDPC treatment. It can be seen that the HEDPC treatment obviously refined the solidification microstructure of the alloy, also with the increase of the pulse current density, the austenitic grain size of the solidification microstructure decreases. In this study the austenitic grain size was refined from 120 μm to 0.6 μm after the HEDPC treatment. The average grain size of each specimen was measured using a standard line from the micrograph of the alloy, counting at least 300 intercepts for each micrograph. The relationship between the austenitic grain size and the pulse current density are shown in Fig. 3.

The start temperature of the HEDPC treatment was very important, which obviously affected the solidification microstructure of the alloy. If the start temperature was higher than the melting point of the alloy, the liquid alloy would splatter, in that case, there were some holes in the solidification microstructure caused by explosion in some micrarea of the liquid alloy. The microstructure is show in Fig. 4. Further, if the start temperature was over higher than the melting point of the alloy, the liquid alloy would explode, in that case, the solidification sample can't be obtained. The suitable start temperature of the HEDPC treatment is at about melting point of the alloy tested.

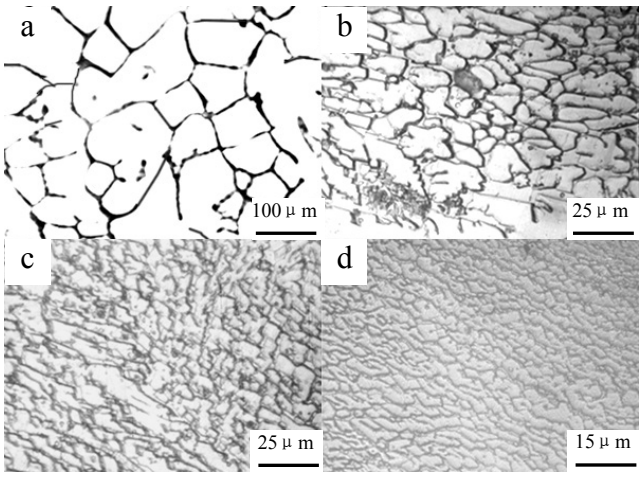

Fig. 2. Solidification microstructures of FeCrNi alloy under different energy density HEDPC treatment: a – 0 A/cm^2; b – 1.1×10^4 A/cm^2; c – 3.1×10^4 A/cm^2; d – 5.2×10^4 A/cm^2

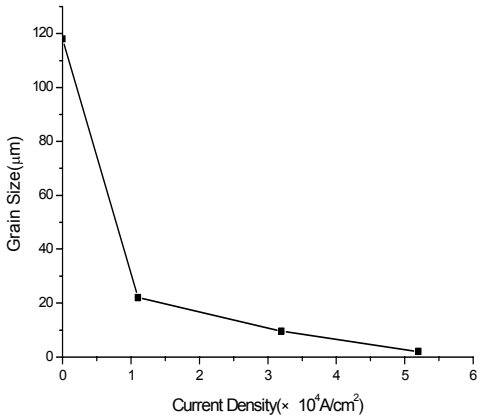

Fig. 3. Relationship between austenitic grain size and pulse current density for the FeCrNi alloy

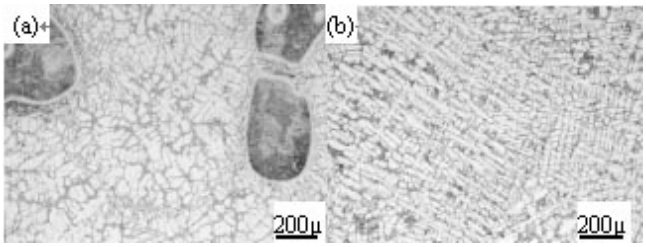

Fig. 4. Solidification microstructures of the alloy subjected to HEDPC treatment under different start temperature at (a) higher than melting point (1500 °C) of the alloy and (b) about melting point (1470 °C)

The reason that the HEDPC treatment refines the solidification microstructure of metal can be explained by the formulation of $dP = BJdL$ built by Nakada [5]. In this equation B is magnetic flux density vertical current line, J is current density, P is pressure and L is distance along current line. It is known from the equation, when pulse current passes through liquid metal, the instantaneous magnetic pressure produced by pulse current is enhanced

with the increase of pulse current density *J*. And when the pulse current density reaches to a certain amount, its instantaneous pressure has far exceeded its internal dynamic pressure, liquid alloy will be compressed over and over again, and make it alternately move vertically to the current direction. Moreover, the value of *B* and *J* differed at different position, which builds up a pressure gradient and form local velocity of flow difference. Finally it shear, so as to impel the dendritic crystal break, thereby, refine the solidification microstructure of metal.

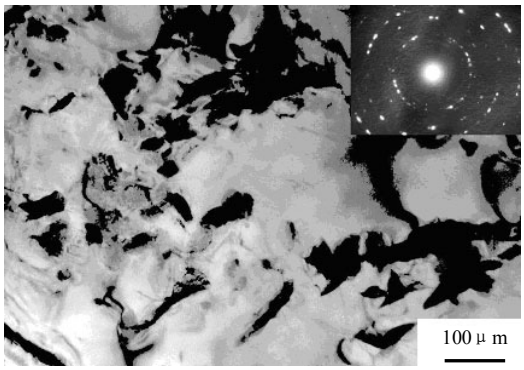

Fig. 5. TEM photo of FeCrNi alloy after HEDPC treatment with current density at 5.2×10^4 A/cm^2

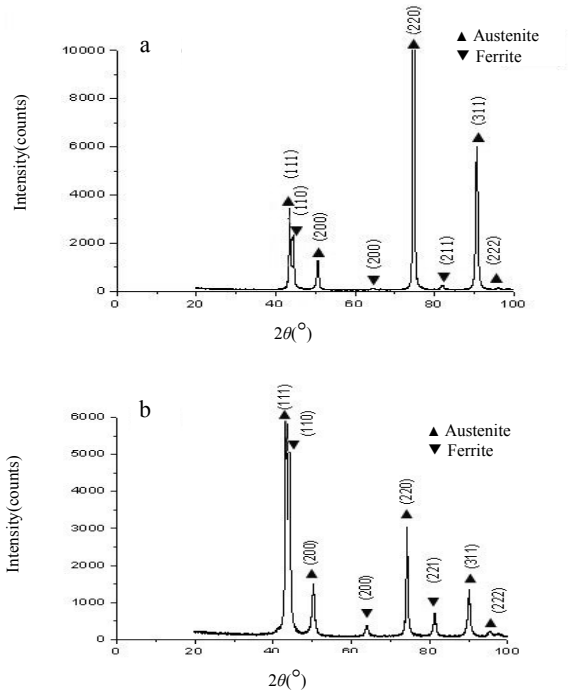

Fig. 6. X-ray diffract spectra of solidification microstructure of FeCrNi alloy: a – 0 A/cm^2; b – 3.1×10^4 A/cm^2

Fig. 5 gives TEM photo of the solidification microstructure of the alloy after the HEDPC treatment. It can be seen that the alloy is composed of two phases, i.e. austenite and ferrite; moreover, the ferrite has been refined to nanometer scale. The X-ray diffraction results of the alloy is shown as Fig. 6, it indicates that the solidification microstructure of the alloy either with or without the HEDPC treatment are austenite-ferrite two phases microstructure, and the austenite phase is as based. But after the HEDPC treatment, the peak positions of austenite and ferrite phases in the X-ray diffraction spectra have been slightly replaced, because the HEDPC treatment may change the distribution of the alloying elements in the alloy [3]. Additionally, it also indicates that the HEDPC treatment changes the relative content of austenite and ferrite in the alloy, the content of the austenite and ferrite phases is 90 % and 10 % without the HEDPC treatment and 81 % and 19 % for the alloy subjected to the HEDPC treatment respectively. It maybe happened because the HEDPC treatment led to the change of the eutectic composition of the alloy, the study on this aspect is still in progress.

CONCLUSIONS

The HEDPC treatment can obviously refine the solidification microstructure of the Fe70Cr18Ni12 alloy, the austenitic grain size of the solidification microstructure can reduce two orders of magnitude to 0.6 μm from 120 μm, the size of the ferrite phase can be refined to nanometer scale.

The suitable start temperature of the HEDPC treatment is at about melting point of the alloy tested.

The HEDPC treatment changed the relative content of the austenite and ferrite phases in the solidification microstructure of the alloy.

Acknowledgments

The work is supported by National Nature Science Foundation of China and Nature Science Foundation of Hebei Province. The project number is 50171057 and 501193 respectively.

REFERENCES

1. **Bottinger, W. J., Coriell, S. R., Greer, A. L.** Solidification Microstructures: Recent Developments, Future Directions *Acta Metallurgica Sinica* 48 (1) 2000: pp. 43 – 70.
2. **Zhang, Z. Z., Song, G. S., Yang, G. C.** Microstructure Characteristics of High Undercooled Bulk Nanocrystalline Fe-B-Si Eutectic Alloy *Acta Metallurgica Sinica* 35 (7) 1999: pp. 693 – 697.
3. **Cui, J. Z.** Solidification of Al Alloys under Electromagnetic Field *Transactions of Nonferrous Metals Society of China* 13 (3) 2003: pp. 473 – 483.
4. **Misra, A. K.** A Novel Solidification Technique of Metals and Alloys: under the Influence of Applied Potential *Metallurgical Transactions A* 16 A (7) 1985: pp. 1354 – 1355.
5. **Nakada, M., Shiohara, Y., Flemings, M. C.** Modification of Solidification Structures by Pulse Electric Discharging ISIJ International 30 (1) 1990: pp. 27 – 33.
6. **Barnak, J. P., Sprecher, A. F., Conrad, H.** Colony (Grain) Size Reduction in Eutectic Pb-Sn Castings by Electroplating Scripta Metall. 32 (6) 1995: pp. 879 – 884.
7. **Takagi, T., Iwai, K., Asai, S.** Solidified Structure of Al Alloys by a Local Imposition of an Electromagnetic Oscillating Force *ISIJ International* 43 (6) 2003: pp. 842 – 848.
8. **Ahmed, S., Mckannan, E. C.** Control of Gamma' Morphology in Nickel Base Superalloys Through Alloy Design and Densification Processing under Electric Field *Material Science & Technology* 10 (11) 1994: pp. 941 – 952.
9. **Li, J. M., Li, S. L., Li, J.** Modification of Solidification Structure by Pulse Electric Discharging *Scripta Metall Mater.* 31 (12) 1994: pp. 1691 – 1694.
10. **Pierre, V., Alan, P., Okamoto, H.** Handbook of Ternary Alloy Phase Diagrams *ASM International Press* 1995: pp. 640 – 700.
11. **Okane, T., Umeda, T.** Eutectic Growth of Unidirectional Solidified Fe-Cr-Ni Alloy *ISIJ International* 38 (5) 1998: pp. 454 – 460.

DOI: 10.5755/j02.ms.26297