Journal of University of Science and Technology Beijing
Volume 10, Number 6, December 2003, Page 35

Semi-solid slurry preparation and rolling of 1Cr18Ni9Ti stainless steel

Weimin Mao, Aimin Zhao, Dong Yun, Leping Zhang, and Xueyou Zhong

Materials Science and Engineering School, University of Science and Technology Beijing, Beijing 100083, China

(Received 2003-01-22)

Abstracts: The preparation and rolling of the semi-solid slurry 1Cr18Ni9Ti stainless steel were researched. The experimental results show that when stirred for 2-3 min under the given test condition, the semi-solid slurry with about 50%-60% (volume fraction) solid and the spherical primary austenitic grains in the size of 100-200 μm can be obtained, and it is easy to be discharged from the bottom little hole of the stirring chamber. The semi-solid slurry of 1Cr18Ni9Ti stainless steel can be rolled into the given plate successfully. However, the solid phase and liquid phase are easily separated in the rolling process, so that the solid primary austenitic grains are concentrated in the center and the liquid phase is near the edge of the rolled plate. The strengths of the plate rolled in the semi-solid state are higher than that of the traditionally repeated hot-rolled plate of 1Cr18Ni9Ti stainless steel.

Key words: 1Cr18Ni9Ti; semi-solid; electromagnetic stirring; rolling

[*This work was financially supported by the National Natural Science Foundation of China (No.59995440).*]

1 Introduction

The semi-solid forming technology of metals or their alloys was invented in the early 70s of the 20th century and this new technology has many advantages [1]. It can reduce the porosity and solidification shrinkage of the formed castings, and the compact castings with higher strength are easily obtained. It can also reduce the composition segregation and improve the property's uniformity. The sophisticated castings can be formed in the semi-solid state because the deformation resistance can be reduced. The die life can be prolonged as the forming temperature is lowered. The production ratio is high and the percentage of rejects is very low. As a result, the semi-solid forming technology of aluminum alloys and magnesium alloys has been successfully used to manufacture many parts of cars and other machine parts [2-5].

M C Flemings and his fellows of MIT prepared the semi-solid slurries or billets of AISI440C, 304, 4340 steels with mechanical stirring equipment, and did the semi-solid die casting experiments and the compact castings were gained [2,6-9]. Kapranos, *et al.*, also produced the semi-solid billet of M2 high speed steel by Ospray or SIMA methods and carried out the forming experiments [10]. Some progress in the continuous semi-solid cast billet of alloys with high melting point by electromagnetic stirrer was achieved by Blazek *et al.* [11]. However, the main reason, for which the technical improvement of the semi-solid die casting process of iron or steel is delayed, is the cracks usually occur on the die surface for the thermal shock of high melting point slurry, so the die life is too short. The direct rolling process of iron or steel slurry should be the most important area because the roller shape is simpler and the slurry can be supplied continuously, and it is possible for the roller to withstand the thermal shock of high melting point iron or steel slurry. In order to meet the need of semi-solid iron or steel rolling, the mechanical stirring, SIMA and Ospray methods are not proper for the preparation of iron or steel slurry. The mechanical stirring chamber and stirrer life are short and the slurry may be easily polluted. The SIMA, Ospray methods are only used for the preparation of semi-solid billets. Therefore, the electromagnetic stirring may be the important method for continuously producing the iron or steel slurry. Up to today, however, the research about the preparation of steels slurry by electromagnetic stirring and their slurry direct rolling has been rarely published in papers. So this paper studied the preparation of 1Cr18Ni9Ti stainless steel semi-solid slurry by electromagnetic stirring and the rolling of the slurry. The semi-solid slurry with about the solid fraction of 50%-60% can be prepared and rolled into plate, which has provided the experience and experiment basis for steels slurry

---

Corresponding author: Weimin Mao, **E-mail:** weiminmao@263.net

rolling process.

2 Experimental

The raw material in the experiments was 1Cr18Ni9Ti stainless steel and its composition (mass fraction in %) is C, 0.1; Si, $\leqslant 1.0$; Mn, $\leqslant 2.0$; P, $\leqslant 0.035$; S, $\leqslant 0.03$; Cr, 17.43; Ni, 10.63; Ti, 0.59. Mass of the steel of 10-20 kg was melted every time with an intermediate frequency coreless induction furnace. The steel melt was deoxidized by Si-Ca powder. The stirring chamber was preheated to 1080°C. The tapping temperature should be controlled to 1600°C so as to further heat the stirring chamber liner. When the liquid steel was poured into the stirring chamber, it was stirred immediately by the electromagnetic field.

The equipment for the preparation of nondendritic semi-solid iron or steel slurry by electromagnetic stirring is shown in **figure** 1. As the superheated liquid 1Cr18Ni9Ti stainless steel was poured into the stirring chamber, it was stirred right away by the electromagnetic field and was then cooled continuously. While the semi-solid melt was stirred, the cooling rate was appropriately controlled so that the semi-solid melt could be maintained between the liquidus temperature T_l and the solidus temperature T_s for enough long time and the slurry of spherical or nearly spherical primary austenitic grains could be gained. When the semi-solid slurry obtained the given solid fraction, the center block bar of the equipment was raised. The semi-solid slurry flowed out of the stirring chamber from the little bottom hole, and then it went on flowing into the predetermined groove between the two hollow rollers with cooling water and finally was rolled into plate.

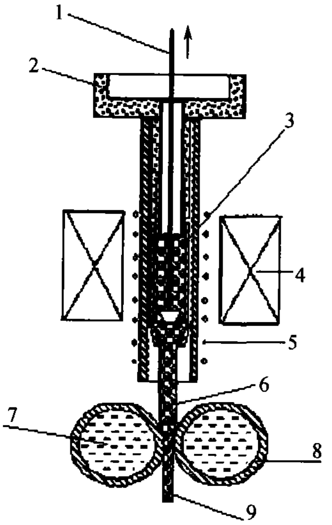

Figure 1 Schematic of the semi-soli steel slurry rolling, 1—center block bar; 2—pouring basin; 3—chamber; 4— electromagnetic stirrer; 5—heating element; 6—slurry;7—cooling water; 8—roller; 9—rolled plate.

In order to roll the semi-solid steel slurry, the slurry preparation parameters should be firstly determined by a series of quenching experiments. A small amount of semi-solid steel slurry was sucked in by a quartz tube and was quenched into a water pool whenever the 1Cr18Ni9Ti stainless steel melt was stirred for a given time. The quenched 1Cr18Ni9Ti stainless steel was made into metallographic samples. The samples were roughly and exactly polished, and then etched by the agent which is composed of 75% (volume fraction) hydrochloric acid and 25% nitric acid. The sample microstructures were observed with the help of an optical microscope so as to analyze the shape and distribution of the spherical primary austenitic grains. The preparation process parameters of the semi-solid 1Cr18Ni9Ti stainless steel slurry were decided from the above experiments. The experimental preparation conditions of the semi-solid 1Cr18Ni9Ti stainless steel by electromagnetic stirring are shown in **table 1**.

Table 1 Preparation parameters of semi-solid 1Cr18Ni9Ti stainless steel by electromagnetic stirring

Sample number	Stirring time / s	Stirring power / kW	Melts number
1	60	9.8	1
2	120	9.8	1
3	180	9.8	1
4	240	9.8	1
5	300	9.8	1
6	60	5.9	2
7	200	5.9	2
8	395	5.9	2
9	440	5.9	2
10	80	4.3	3
11	125	4.3	3
12	480	4.3	3
13	540	4.3	3

The semi-solid slurry rolling experiments were conducted on the designed equipment. The mill is irreversible single-groove mill machine with two hollow rollers cooled by the inner water. The rollers are made of cast iron. The rollers are 240 mm in diameter and the rolling groove between the two rollers is 5 mm wide. The rollers surface temperature was about 50°C when rolling and their rolling speed was 1173 mm/min.

The tensile samples were cut from the steady lengthwise part of the rolled plate and the mechanical properties in ambient temperature were determined. The microstructures of the rolled plate were examined.

3 Results and discussion

3.1 Preparation of the semi-solid slurry

The austenitic grains of 1Cr18Ni9Ti stainless steel will solidify to dendrites under the traditional condition and the austenitic dendrites are very large, as shown in **figure 2**. However, they will solidify to spherical grains and be fined under the condition of electromagnetic stirring, as shown in **figure 3**. The larger spherical particles in figure 3 are the primary austenitic grains before quenching while the fine grains are origin liquid quenched. From figure 3 it can be seen that the electromagnetic stirring power is one of the important parameters. If the stirring chamber is preheated to 1080°C and the pouring temperature is 1600°C, the solidified microstructures are changed completely under the condition of stirring power 9.8 kW and the primary austenitic grains are turned to spherical particles with almost the same size and roundness, as shown in figure 3(a) and (b). After stirred for 2 to 3 min, the primary austenitic shape is almost spherical and the slurry solid fraction is about 50%-60% (volume fraction). When the center block bar of the stirring equipment is lifted at this time, the semi-solid slurry easily flow out of the chamber from the little bottom hole and then goes into the rolling groove between the two rollers. The slurry solid fraction will continue to increase and more than 60% if stirred for more time. When this slurry flows out of the chamber, some slurry may be stuck on the chamber inner surface because the semi-solid slurry is too thick. As a result, the proper process parameters for the preparation of the semi-solid 1Cr18Ni9Ti stainless steel are as following: the chamber temperature should be preheated to 1080°C, and the pouring temperature should be 1600°C; the stirring power should be 9.8 kW and the stirring time should be 2-3 min.

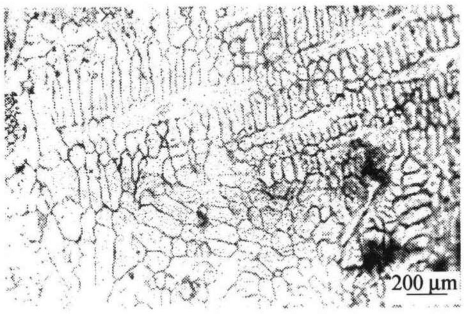

Figure 2 Microstructures of 1Cr18Ni9Ti stainless steel traditionally solidified.

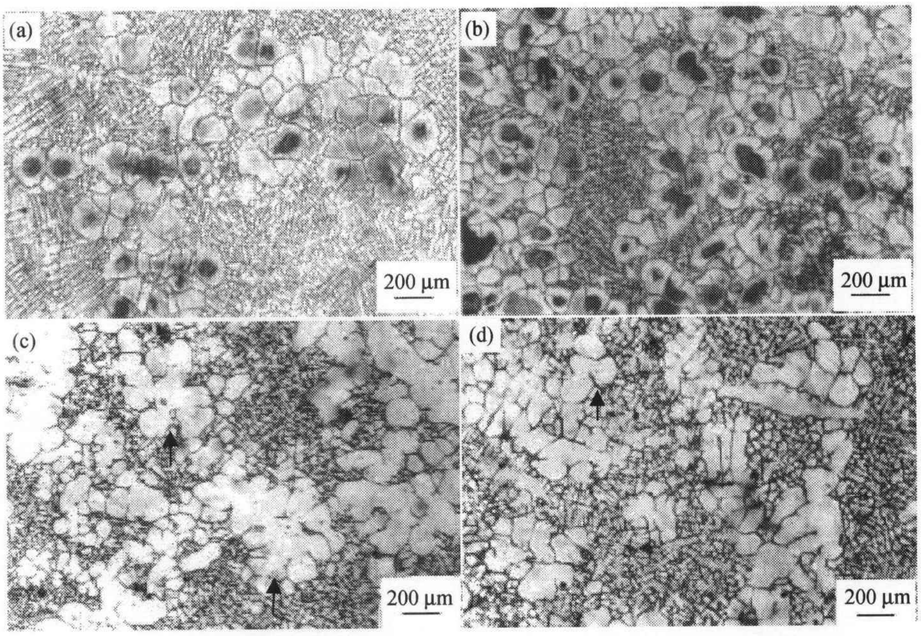

Figure 3 Microstructures of semi-solid 1Cr18Ni9Ti stainless steel under electromagnetic stirring with different powers, (a) sample 2; (b) sample 3; (c) sample 8; (d) sample 9.

If the preheated chamber temperature and the pouring temperature are not changed, and if only the stirring power is dropped to 5.9 kW, the shape of the primary austenitic grains is not ideal although the steel melt also solidifies in stirring. Not only many spherical austenitic grains exist in the microstructures but also many rose-like ones appear which like the equiaxed dendritic grains. The rose-like austenites will not disappear even with the longer stirring time, as shown with the arrows in figures 3(c) and (d). If the stirring power further drops down to 4.3 kW, the shape of the primary austenitic grains is worse and the primary austenitic grains often agglomerate into a big mass, and even the large primary austenitic dendrite exists.

3.2 Roll of the semi-solid slurry

The semi-solid slurry with different solid fractions of 1Cr18Ni9Ti stainless steel was rolled into a plate of 120 mm wide and 5 mm thick. The surface quality of the rolled plate is generally good. However, the slurry will pile up on the roller top and can flows away from the rectangular groove if the slurry is discharged too fast from the stirring chamber, so that the burrs in the direction of width often appear. The microstructures of the plate cross section were detected as shown in figure 3. **Figure 4** shows that the originally spherical austenitic grains and liquid phase are separated and accumulated to some extent in the rolled plate. That is, the spherical austenitic grains are distributed in the center area and the liquid phase is around the periphery of the rolled plate. The microstructure segregation in the rolled plate always exists no matter how much or little the solid fraction of the slurry is. The lower the original solid fraction is, the more serious the liquid segregation exists in the plate. The microstructure segregation in the rolled plate is decreased along with the solid fraction increase. If the solid fraction of the 1Cr18Ni9Ti stainless steel slurry is low, the shape of the spherical austenitic grains is not changed upon rolling. However, if the solid fraction of the 1Cr18Ni9Ti stainless steel slurry is high, the shape of the spherical austenitic grains is changed upon rolling.

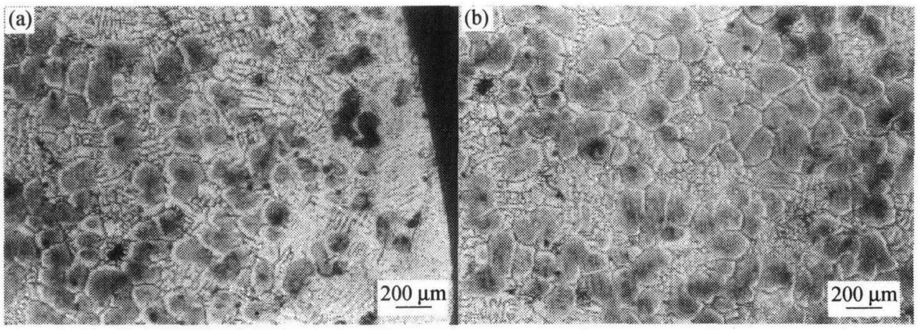

Figure 4 Microstructures of semi-solid rolled plate of 1Cr18Ni9Ti stainless steel, (a) near the edge of the plate; (b) in the center of the plate.

Before the semi-solid slurry of 1Cr18Ni9Ti stainless steel was rolled, it was transported from the stirring chamber. The diameter of the discharged slurry is about 16 mm, but the rolling groove width is only 5 mm, so that the slurry is compressed and fills in the rectangular groove. Because the rolled plate blocks up the groove from the bottom, the slurry is obliged to flow along the shaft direction of the rollers and is formed to the plate. Upon rolling, the flowing resistance of the spherical solid austenitic grains and the liquid phase are different. The viscosity of the liquid phase is lower and the flowing resistance is small, so the liquid in the inner area of the slurry would easily flow out through the little gap among the solid grains. The spherical solid austenitic grains would collide each other on flowing and the flowing resistance is high, so the flowing speed is slower than the liquid and the phenomena of solid and liquid separation emerges after rolled. The lower the linear speed of the rollers is, the longer the flowing time of the liquid and the more serious the segregation are, so the semi-solid slurry should be rolled in a proper speed and a proper solid fraction.

3.3 Mechanical properties of the rolled plate

The tensile samples of 1Cr18Ni9Ti stainless steel were cut from the steady lengthwise part of the rolled plate as shown in **figure 5**. The tension experiments were completed at room temperature and the results are shown in **table 2**. Compared with the properties of the traditionally rolled 1Cr18Ni9Ti stainless steel, the strengths of the plate rolled in the semi-solid state is increased somewhat, but the elongation is decreased. The reasons may be that the microstructures in the rolled plate are not homogeneous and the rolling times are not enough. If some process measures are taken to reduce the microstructure segregation in the rolled plate, it is probable to promote the homogeneity of the mechanical properties.

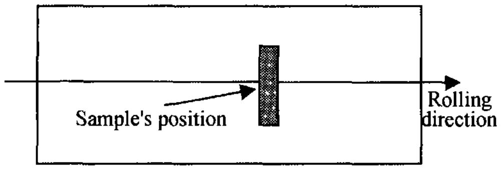

Figure 5 Position of the draw samples cut from the rolled plate.

Table 2 Properties comparison between the semi-solid rolled plate and traditional hot-rolled plate of 1Cr18Ni9Ti stainless steel

Rolling process	Heat treatment	Rupture strength / MPa	Yield strength/MPa	Elongation / %
Traditional repeated rolling	Quenching at 1100°C	541-790	196-510	40-81
One single-time semi-solid rolling	Quenching immediately after rolled	766	492	27.7

4 Conclusions

(1) The proper process parameters for the preparation of the semi-solid 1Cr18Ni9Ti stainless steel slurry are as following: the chamber temperature should be preheated to 1080°C, and the pouring temperature should be 1600°C; the stirring power should be 9.8 kW and the stirring time should be 2-3 min. The diameter of spherical or near spherical primary austenitic particles is about 100-200 μm, and the solid fraction is about 50%-60% (volume fraction) under the above conditions and this slurry easily flows out of the chamber.

(2) The semi-solid slurry of 1Cr18Ni9Ti stainless steel was successfully rolled to a plate, but the originally spherical primary austenitic grains were separated with the original liquid after rolled in some extent, and the spherical solid grains were distributed in the center area and the liquid phase was around the periphery of the plate. Moreover, the lower the solid fraction is, the more serious the liquid segregation is.

(3) Compared with the properties of the traditionally rolled 1Cr18Ni9Ti stainless steel, the strengths of the plate rolled in the semi-solid state is increased somewhat, but the elongation is decreased.

References

[1] D.B. Spencer, R. Mehrabian, and M.C. Flemings, Rheological behaviour of Sn-15pct Pb in the crystallization range [J], *Metall. Trans.*, 3A (1972), p.1925.

[2] M.C. Flemings. Rheological behaviour of Sn-15pct Pb in the crystallization range [J], *Metall. Trans.*, 22A(1991), No.5, p.957.

[3] M. Garat and S. Blais, Aluminium semi-solid processing:from the billet to the finished part, [in] A.K Bhasin, J.J Moore, K.P. Young, and S. Midson eds: *The 5th Int. Conf. on Semi-Solid Processing of Alloys and Composites, Golden, Colorado* [C]. USA: The Colorado School of Mines, Colorado, 1998: xvii-xxxi.

[4] P. Eisen and K. Young, Diecasting systems for semiliquid and semisolid metalcasting-applications, [in] G.L. Chiarmetta and M. Rosso eds: *The 6th Int. Conf. on Semi-Solid Processing of Alloys and Composites* [C]. Italy: The Department of Materials Science and Chemical Engineering, Politecnico Di Torino, 2000, p.41.

[5] W.M. Mao, A.M. Zhao, Y.J. Li, *et al.*, Temperature field and microstructural formation of semi-solid $AlSi_7Mg$ alloy [J], *J. Univ. Sci. Technol. Beijing*, 7(2000), No.2, p.99.

[6] J.M. Oblak and W.H. Rand. Solid-liquid phase characterization of several rheocast high performance alloys [J], *Metall. Trans. B*, 7B(1976), No.12, p.699.

[7] J.M. Oblak and W.H. Rand, Structure and properties of the rheocast ferrous alloy 440C [J], *Metall. Trans. B*, 7B(1976), No.12, p.705.

[8] D.A.V.Cleave. Stirring action opens up steel die casting [J], *Iron Age*, 220(1977), No.8, p.34.

[9] K.P. Young, R.G. Riek, and M.C. Flemings, Structure and properties of thixocast steels [J], *Met. Technol.*, 6(1979), No.4, p.130.

[10] P. Kapranos, D.H. Kirkwood, and C.M. Sellars, Thixoforrming high point alloys into non-metallic dies., [in] D. H. Kirkwood and P. Kapranos eds: *The 4th Int. Conf. on Semi-Solid Processing of Alloys and Compositions* [C]. UK: The Department of Engineering Materials, University of Sheffield, 1996, p.306.

[11] K.E. Blazek, J.E. Kelly, and N.S. Pottore, The development of a continuous rheocaster for ferrous and high melting point alloys [J], *ISIJ Int.*, 35(1995), No.6, p.813.