Influences of different degassing processes on refining effect and properties of 4004 Al alloy

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Abstract: In order to improve the plasticity of 4004 Al alloy and subsequently the productivity of 4004 Al foil, the research studied in detail the influence of the rotary impeller degassing process on the refining effect of 4004 Al alloy, in which the impacts of four major parameters: gas flow, rotational speed, refining time, and stewing time, on degassing rate of 4004 Al alloy was systematically studied by using an orthogonal experiment methodology. Results show that the rotational speed has the greatest impact on the degassing of 4004 Al alloy, followed by gas flow and refining time; stewing time has the least impact. The optimum purification parameters obtained by current orthogonal analysis were: rotor speed of 500 $r \cdot min^{-1}$, inert gas flow of 0.4 $mL \cdot h^{-1}$, refining time of 15 min, and stewing time of 6 min. Degassing rate using the optimum parameters reaches 68%.

In addition, the comparison experiments among C_2Cl_6 refining, rotary impeller degassing, and combined treatment of C_2Cl_6 refining and rotary impeller degassing for 4004 Al alloy were performed. The experimental data indicated that the combined treatment of C_2Cl_6 refining and rotary impeller degassing has the best degassing effect. Degassing rate of C_2Cl_6 refining, rotary impeller degassing and combined refining treatment is 39%, 69.1% and 76.9%, respectively. The mechanical properties of the specimen refined by rotary impeller degassing were higher than those by C_2Cl_6 refining, but lower than those by combined refining treatment.

Key words: aluminum alloys; refinement technology; rotary impeller degassing

CLC numbers: TG146.21 **Document code:** A **Article ID:** 1672-6421(2013)02-104-04

4004 Al alloy has been widely used as brazing foil in automobile, air conditioner, oxygen producer, and so on [1, 2]. In the production of 4004 Al, the high gas content results in a loose microstructure, which leads to edge cracks and strip break during the rolling process [3]. The refining of Al alloy melt is one of the key processes to improving the properties of 4004 alloy[4]. Chlorides have been widely used as refining agents in production, however the reproducibility is low and the degassing rate is affected by both the chloride types (C_2Cl_6, $MnCl_2$, and $ZnCl_2$) and practical operation of workers. Further, a large amount of toxic chloride gas is produced during the process, which pollutes the environment, threatens the health of the operators, and seriously corrodes equipment [5]. Nowadays, mechanical refining (using high pure argon or nitrogen as refining agent) has been widely used due to the better refining effect and lower environment pollution [6-8]. The mechanical refining effects of many aluminum alloys have been well studied. Yet, little has been reported on the rotary impeller degassing for 4004 aluminum alloy.

In this paper, the relationships among degassing rate and gas flow, rotational speed, refining time, and stewing time during the rotary impeller degassing for 4004 Al alloy were investigated in detail. An optimum process was obtained by the orthogonal analysis. Degassing effect using C_2Cl_6 refining agent and a combined refining technique was also evaluated.

1 Experimental detail

1.1 Rotary impeller degassing

The rotary impeller degassing was carried out on XD-J-100BP dehydrogenation machine using high pure argon (99.999%) with a pressure of 0.5 MPa. An orthogonal L9 (3^4) test was designed to study the influence of four parameters such as gas flow, rotational speed, refining time and stewing time (listed in Table 1) on the degassing effect. The hydrogen concentration detector (NOTORP KYHS-A2, Japan) was used to measure gas content in Al melts.

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Received: 2012-04-16 Accepted: 2012-12-15

Table 1: Processing parameters used in the orthogonal experiment

	Factors			
	Gas flow $m^3 \cdot h^{-1}$	Rotational speed $r \cdot min^{-1}$	Refining time min	Stewing time min
1	0.2	300	5	3
2	0.3	400	10	6
3	0.4	500	15	9

4004 Al alloy was melted at 720 ℃ in an electric resistance furnace. The gas concentration of the melt was measured before and after refining treatments. The optimized parameters were obtained through an orthogonal analysis. The tensile properties of as-cast 4004 alloy were also examined.

1.2 Combined treatment of C_2Cl_6 refining and rotary impeller degassing

Hydrogen concentration of melt was measured after the melt temperature reached about 720 ℃. Then C_2Cl_6 mixed refining agent (C_2Cl_6 of 75% and Na_2SiF_6 of 25%, with a total of 0.6wt.% of the melt) was added into the melt. After refining 10 min and stewing 10 min, the melt was heated up to about 720 ℃ to further refine using a rotary impeller. After the refining process was completed using the optimum parameters, hydrogen concentration of melt was measured again. The tensile properties of samples refined by combined refining treatment were also tested.

2 Results and discussion

2.1 Orthogonal analysis of rotary impeller degassing process

An Orthogonal L9 (3^4) test was designed to optimize the rotary impeller degassing process. Orthogonal array is a fractional factorial design, in which orthogonal array was used to assign process parameters to a series of experimental combinations. The results were then analyzed using a common mathematical procedure (for example extreme difference analysis). Although various parameters potentially affect the refining effect, the gas flow, rotational speed, refining time and stewing time are generally considered as the major factors. Optimization of degassing process can be carried out by using an orthogonal L9 (3^4) test design. The meanings of all numbers in L9 (3^4) were explained in detail in Ref. [9].

Hydrogen concentrations in 4004 Al alloy processed by rotary impeller degassing method were measured. The degassing rate was calculated and summarized in Table 2.

Table 2: Hydrogen concentration of samples refined using rotary impeller degassing method

N0.	Factors				Hydrogen concentration $mL \cdot kg^{-1}$		Degassing rate (%)
	A–Gas flow $m^3 \cdot h^{-1}$	B–Rotational speed $r \cdot min^{-1}$	C–Refining time min	D–Stewing time min	Before refining	After refining	
1	0.2	300	5	3	2.19	1.60	26.94
2	0.2	400	10	6	2.28	1.29	43.42
3	0.2	500	15	9	2.08	0.93	55.29
4	0.3	300	10	9	2.10	1.32	37.14
5	0.3	400	15	3	2.21	0.97	56.11
6	0.3	500	5	6	2.24	0.75	66.52
7	0.4	300	15	6	2.31	1.19	48.48
8	0.4	400	5	9	2.31	0.92	60.17
9	0.4	500	10	3	2.30	0.72	68.70

It is shown from extreme difference analysis of degassing rate (Table 3.) that the rotational speed of rotor is the most important factor, and the importance is reduced in the order of gas flow, refining time and then stewing time. The combination of the best refining parameter obtained is $B_3A_3C_3D_2$, which refers to the setting of the rotational speed of 500 $r \cdot min^{-1}$, inert gas flow of 0.4 $mL \cdot h^{-1}$, refining time of 15 min, and stewing time of 6 min.

Table 3: Extreme difference analysis of degassing rate

Factors	Sum of level 1 I	Sum of level 2 II	Sum of level 3 III	Degree	Best combination
A– inert gas flow (Q), $m^3 \cdot h^{-1}$	41.88	53.26	59.12	17.24	2
B–rotational speed, $r \cdot min^{-1}$	37.52	53.23	63.50	25.98	1
C– purification time, min	51.21	49.75	53.29	3.54	3
D–stewing time, min	50.58	52.81	50.87	2.23	4

The tensile properties of the degassed 4004 alloy are listed in Table 4. For comparison, the tensile strength and elongation of 4004 alloy without purification were tested as well; they are 163 MPa and 3.6%, respectively. The direct comparison strongly proves that the rotary impeller degassing can effectively improve the tensile properties of 4004 alloy. The tensile strength and elongation of 4004 alloy increase with the increase of the degassing rate. The reason is that with the increase of degassing rate, the hydrogen concentration in 4004 alloy decreases, and the number of pores in the alloy also decreases. As a result, the tensile properties, especially the elongation of the alloy, are improved.

Table 4: Tensile properties of 4004 Al alloy after refining

No. of sample	1	2	3	4	5	6	7	8	9
Tensile strength MPa	166	176	181	173	182	190	179	185	194
Elongation %	5.0	5.3	5.4	5.1	5.4	5.5	5.3	5.5	5.6

2.2 Degassing effect of different refining methods

Degassing results of the samples refined by different methods are listed in Table 5. It is shown that the degassing rate of combined refining treatment is higher than that of rotary impeller degrassing, and almost doubles that of C_2Cl_6 refining.

Table 5: Degassing results of different refining processes

Refining method	Hydrogen concentration (mL·kg^{-1})		Degassing rate (%)
	Before refining	After refining	
C_2Cl_6 and rotary impeller combined refining	2.21	0.51	76.9
C_2Cl_6 refining	2.18	1.33	39.0
Rotary impeller degassing	2.23	0.69	69.1

The tensile properties of 4004 Al alloy refined by different methods are listed in Table 6. The results show that samples refined by combined refining method have the best tensile properties in both strength and ductility. Samples refined by rotary impeller degassing method have better tensile properties than those refined solelyby C_2Cl_6 refining.

Table 6: Tensile properties of refined 4004 Al alloy

Method	Rotary impeller degassing	C_2Cl_6 refining	Combined refining
Tensile strength (MPa)	193	187	196
Elongation (%)	5.6	4.5	6

Figure 1 shows typical as-cast microstructure and the porosity distribution in 4004 alloy refined by different processes. Pores are noted as white arrows in Fig.1. It is obvious that the number of pores in degassed 4004 alloy is much less than that of 4004 alloy without any degassing treatment. Sample refined by the combined refining method

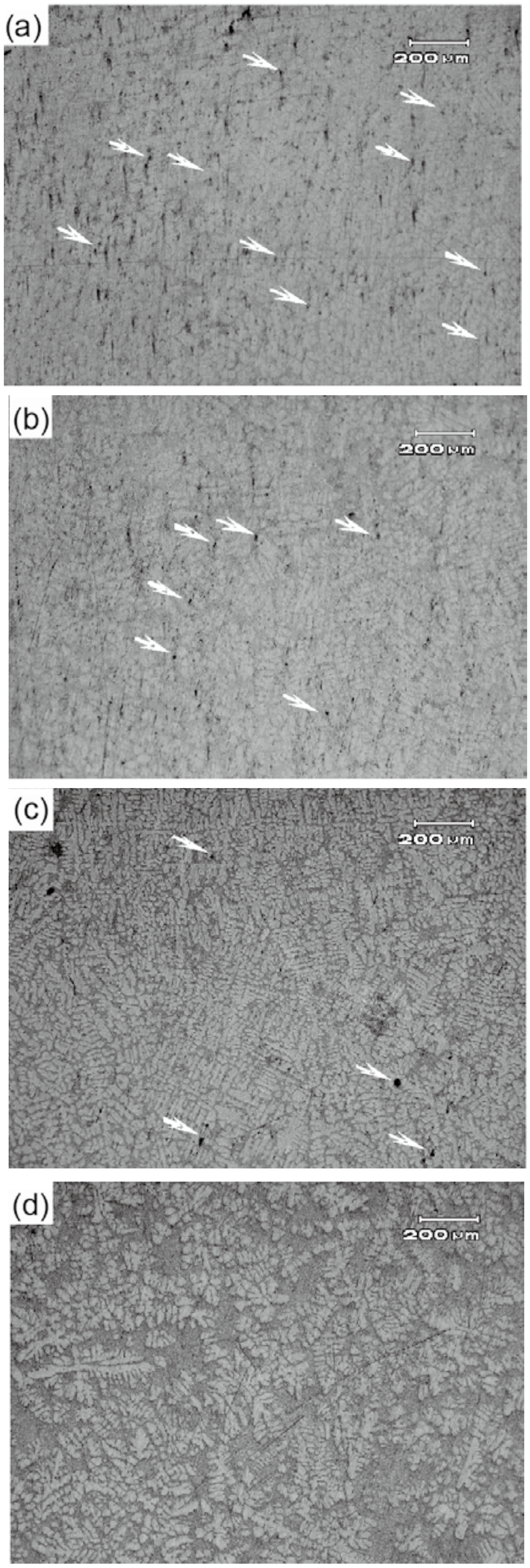

Fig. 1: Pore distributions of samples refined by different methods: no refining (a), C_2Cl_6 refining (b), rotary impeller degassing (c), and combined refining (d)

has the densest microstructure. Sample refined by rotary impeller degassing method is denser than the sample refined by C_2Cl_6. Since the existence of pores decreases loading area and the continuity of the alloy, the tensile properties of the alloy will be undermined by the pores. In summary, an optimum degassing processing technique is highly desired for a better degassing effect and therefore better tensile properties.

3 Conclusions

(1) An orthogonal experiment was designed, performed and analyzed. The results show that the rotational speed is the most significant parameter that influences the degassing rate of rotary impeller degassing, and then the significance reduces in the order of gas flow, refining time and stewing time. The optimum process parameters (corresponding to a degassing rate of 69.1%) were determined from this study as rotational speed of 500 $r \cdot min^{-1}$, gas flow of 0.4 $mL \cdot h^{-1}$, refining time of 15 min, and stewing of 6 min.

(2) For 4004 Al alloy degassed by rotary impeller, the tensile properties, especially elongation, increase with the increase of degassing rate.

(3) Degassing effect of rotary impeller and C_2Cl_6 combined refining is better than that of rotary impeller degassing. The degassing rate can reach up to 76.9% with a hydrogen concentration as low as 0.51 $mL \cdot kg^{-1}$.

(4) The tensile properties of Al samples produced by rotary impeller degassing are higher than that produced by C_2Cl_6 refining only, but a little bit lower than that produced by the combined refining treatment.

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This work was financially supported by the Heilongjiang Province Natural Science Foundation (ZD20081901), and the Foundation of Heilongjiang Educational Committee (11541051).